CYCLIC VACUUM CAVITATION FOR CLEANING APPLICATIONS

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A Thesis

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Sam Houston State University

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In Partial Fulfillment

of the Requirements for the Degree of

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by

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DEDICATION

I would like to dedicate my thesis to my greatest research advisor, Dr. Darren L. Williams, my parents, my loving and caring husband, Sakitha Ariyarathne, and all the teachers who taught me and helped me to enhance my academic knowledge.

ABSTRACT

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The removal of micron-sized carbon black particles from capillary tubes (outer diameter 1.46 mm, inner diameter 1.12 mm, length 50.00 mm) using cyclic vacuum cavitation (VC) cleaning was investigated. As the first objective of this study, the VC apparatus was constructed. This VC setup allowed video monitoring of the cleaning process. Photographs, video recordings, and gravimetric analysis tests were used to identify the cleaning efficiencies. As the second part, the carbon black powder removal efficiencies were used to compare cyclic VC and ultrasonic cavitation (UC) cleaning. Cyclic VC coupled with deionized (DI) water was able to remove 67 ± 7% of carbon black powder from contaminated capillary tubes. Solutions of 1% sodium lauryl sulfate (SLS) in DI water successfully removed 82 ± 7% of carbon black powder using fifteen vacuum cycles at room temperature. The UC process was unable to flush the carbon black soil from the internal volume of the capillary tubes. The cleaning was more effective with cyclic VC than UC cleaning at given conditions. Raising the temperature, of the liquid increased cavitation and caused better wetting of the carbon black powder at the bottom of the tubes. The optimized VC process parameters (25 vacuum cycles, 1% SLS in DI water, vigorous stirring at 40 °C) removed 99 ± 1% of the carbon black powder. This study showed experimental evidence that cyclic VC is a good cleaning approach for cleaning parts that have deep blind holes.

KEYWORDS: Cyclic vacuum cavitation cleaning, Ultrasonic cleaning, VC apparatus, Capillary tubes, Carbon black powder removal

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CHAPTER I

Introduction

The overall objective of this thesis is to provide a detailed study of the cyclic vacuum cavitation (VC) cleaning method. Cyclic VC is a promising new method to clean components with blind holes and complex geometries. This study will first determine if a small lab-scale apparatus can be built to clean soiled capillary tubes. Exposure to a vacuum can form cavities inside a cleaning fluid. These vacuum-generated cavities help to gently remove soil particles from the soiled components. The generation and dissolution of cavities can be cycled as much as needed to produce a very effective and very gentle cleaning action. Capillary tubes are an extreme example of what is called blind holes in manufacturing. Blind holes are very difficult to clean. For example, ultrasonic cleaning has difficulty removing contaminants within blind holes or tubes since ultrasonic cavitation does not flush internal surfaces effectively. Therefore, the second goal of this study is to document the performance differences between ultrasonic cavitation and vacuum cavitation methods with capillary tubes.

Early studies of the cyclic vacuum cavitation process were performed by Donald Gray and Richard W. Plavidal.[1,2] Plavidal and Gray called this technique “vacuum cycle nucleation”, and others call this the “cyclic nucleation process”.[3] They include the word nucleation in their description of the method which emphasizes the initiation of cavities in the cleaning fluid. However, we have chosen to call this technique “vacuum cavitation” cleaning to emphasize the phenomenon that causes the cavitation. Our phenomenon-based terminology is similar to the use of ultrasonic to describe the phenomenon causing cavitation in ultrasonic cavitation cleaning. Plavidal was able to

create a dynamic chamber for vacuum cavitation.[2] In this chamber, the chamber volume can be cyclically enlarged and reduced. It will then reduce and increase the chamber pressure.

Fei Chen et al. used vacuum-generated cavities to clean bacterial spores from a series of surrogate spacecraft materials using H_2O_2 as the cleaning fluid at 60 °C.[4] This method completely cleaned the bacterial spores with a 99.90 confidence level. Since H_2O_2 was considered as a sporicidal agent, Fei Chen and group needed to show that the spores were removed from the sample surfaces. Even though highly concentrated H_2O_2 cleaning fluid was used, it did not show any significant sporicidal activity at the temperature used in the treatment. This leads them to conclude that spores were not killed and left behind but were actually removed from the sample surfaces.

Zhang Jun et al. discussed vacuum vapor phase cleaning to remove the soil from the bottom side of ball grid arrays (BGA) circuit packages.[5] The cleaning of electronic parts has become difficult due to their complex structures. Modern BGA chips feature gaps in the tens of microns between the chip and the circuit board. After the soldering processes, electronic products can be contaminated with flux residues and other soils. Using vacuum cycle cleaning, solvent can easily enter the gaps beneath BGA chips. Therefore, Zhang Jun and group were able to clean these parts without damaging the solder joints.

This technology has been implemented in the cleaning industry by Vacuum Processing Systems (www.vacuumprocessingsystems.com) and LPW Reinigungssysteme GmbH (www.lpw-reinigungssysteme.de). However, our objective was to document the performance of cyclic VC cleaning in an independent academic study.

Cleaning and surface properties

Selecting a cleaning process for soiled components is a vital step in the manufacturing industry. This selected cleaning process should be fast, efficient and cause the least possible damage to the parts while removing the target contaminant from the surface of the soiled components.[6] Usually, the cleaning techniques are strongly influenced by a range of factors, such as the nature of the soil, substrate properties, cleaning facilities, cost for cleaning, the degree of cleanliness, environmental impact, and the effect of the previous process on the component. Likewise, soils can make chemical and physical interactions with the surface of the components.[7] Thus, understanding component–soil interactions is an important step in cleaning.

Basic processes like cleaning with solvents, cleaning with detergents, chemical reactions and mechanical forces are commonly considered in the parts cleaning industry. Adding a solvent or aqueous solution to dissolve the soil from a soiled component's surface is the most frequently used method in the cleaning industry. The solvent or detergent solution detaches soils from the surface and carries the soil away by flow, spray, agitation, draining, or rinsing. The addition of a chemical to cause a chemical reaction to convert soil to another form so that it can be removed easily is also a common method of the cleaning industry. Mechanical cleaning is done by pushing, scraping, sucking, blowing, beating, rubbing, or physically moving dirt from a component. In addition to these common methods, other complex methods exist such as vapor degreasing, laser cleaning, and glow discharge methods.

Types of soil

In practice, the amount of soil adsorbed and the adsorption strength will vary widely for different soils because of different soil-surface interactions. Thus, for some strongly adsorbed soils, it is evident that significant amounts of soil will remain on the surface even after extensive cleaning. Different types of soil and their cleaner types are listed in Table 1. They can be categorized as organic, inorganic, petroleum and combination soils.

Table 1. Types of soil and their cleaner types.

Soil type	Examples	Cleaner type
Organic	Living matter, food	Alkaline aqueous
Inorganic	Rust, minerals	Acid aqueous
Petroleum	Oil, grease	Petroleum solvents and/or aqueous detergents
Combination		Combinations of the above

Generally, alkaline or basic aqueous cleaners are used to remove organic soils from the components. Inorganic soils can be identified as minerals, scale, or rust. To remove them, acidic aqueous cleaners are commonly used. Petroleum soils are typically oils and greases. They can be removed using petroleum-type solvents and also alkaline aqueous cleaning solutions. Of course, soil can be a combination of all these soil types. To clean these kinds of soils, a combination of different types of cleaners can be used. Furthermore, soil can be divided into polar and nonpolar depending on its water solubility (Figure 1).

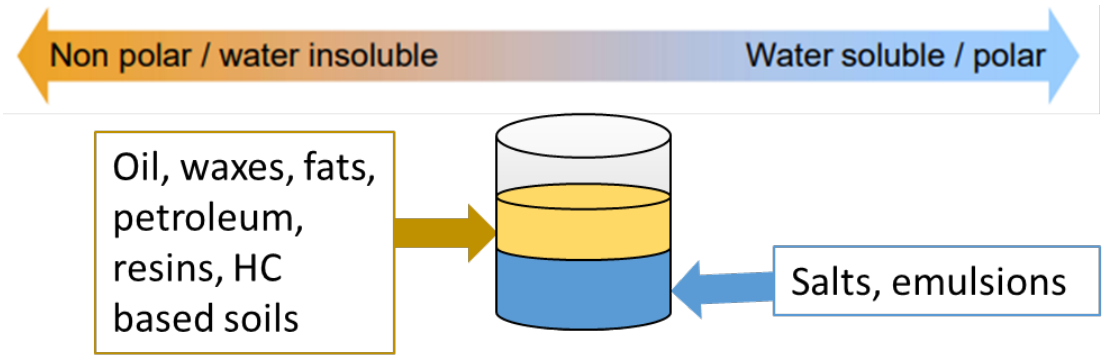

Figure 1. Nature of the soil.

Boundary layer

Cleaning the soil from the surface frequently involves the use of cleaning fluid. Therefore, the properties of fluid-surface interactions are important when trying to remove soils from the surface. The fluid region near the surface is the laminar flow region which moves very little. There is a boundary where the fluid changes from being a surface-interacting laminar fluid to a bulk, freely-moving fluid which exhibits turbulent flow. The laminar flow layers can be seen close to the surface in Figure 2. Ultrasonic cavitation takes place in the bulk fluid and has some difficulty interacting directly with the surface because of the boundary layer of surface fluid. Micro jets of collapsing (transient) ultrasonic cavities can shoot through this boundary layer, but these same jets can damage the surface of the part (Figure 3). Vacuum-generated cavities form at the surface so they are by definition inside the boundary layer of the fluid.

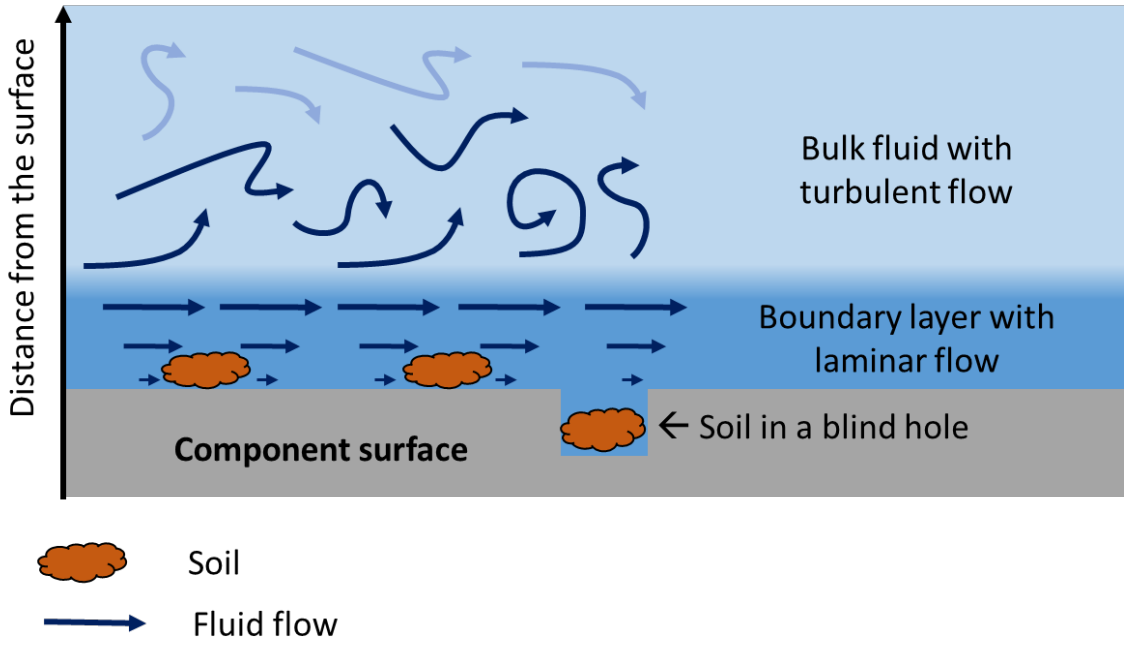

Figure 2. Boundary layer diagram.

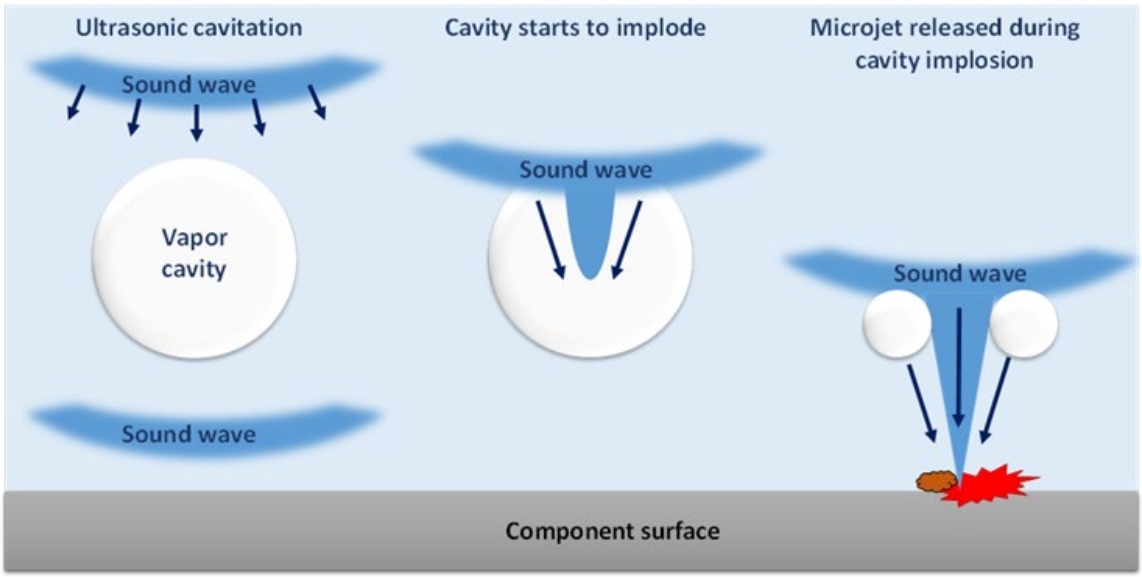

Figure 3. The implosion of transient vapor cavities in ultrasonic cleaning.

Cleaning methods

Though there are many cleaning methods, this section only discusses cleaning with solvents, cleaning with detergents, and cavitation cleaning.

Cleaning with solvents

The interactions between the soil and the solvent will determine whether the soil will dissolve in the solvent. If the soil-surface interaction is strong compared to the soil-solvent interaction, dissolution of soil will not happen. Otherwise, the soil will dissolve in the solvent. To determine this phenomenon, Hansen Solubility Parameters (HSPs) can be used. Hansen solubility parameters were created by Charles M. Hansen in 1967 as part of his Ph.D. thesis. They were developed through the like-dissolves-like properties of the solvents.[8] Molecular interactions between soil and solvent depend upon the chemical species of the soil and solvent. After determining the species in the soil, HSP solvent blends can be applied to the cleaning process. Furthermore, the solubility of non-polar hydrocarbon molecules in water, such as oils and greases, is very low because they cannot make hydrogen bonds with water. Therefore, water alone cannot clean non-polar molecules. Alcohols are commonly used for cleaning applications. They can interact with both polar and non-polar molecules. The strength of the interaction depends on the aliphatic chain length. Also, they can form hydrogen bonds. Therefore, they can dissolve various chemical species. Their main disadvantages are flammability and toxicity. Halogenated solvents are very effective in dissolving many types of greases, but some are toxic.[7] Therefore, before selecting a solvent for the cleaning application, it is crucial to consider the nature of the soil and solvent, toxicity and flammability of the solvent.

Cleaning with detergents

A detergent is a blend of surfactant and water. A surfactant is a surface-active agent, a substance that can reduce the surface tension of water through preferential association with the air-liquid interface. Surfactants consist of a hydrophilic head group

and a hydrophobic tail group. Therefore, they can associate with both hydrophilic and hydrophobic chemical groups.[7] Typically, the cleaning mechanism of a surfactant can be twofold. At first, the surfactant reduces the surface tension of the aqueous cleaning solution allowing water to flow into small spaces that pure water would avoid. As well, it lowers the surface energy of the substrate avoiding re-adsorption of the contamination.[7] The contact area of the contaminant with the surface is decreased and removal of soil through agitation becomes straightforward. Secondly, the contaminant may be surrounded by the surfactant and captured in a micelle. Recontamination is avoided as the micelle is dispersed in the solvent.[9]

Cleaning with cavities

Cavities are well-known for their cleaning potential.[10] They can be generated by sound waves, lasers, reduced pressure, or other techniques. Cavitation is defined as the formation of void space within a fluid. Cavities are different from bubbles. Bubbles are identified as a pocket of air surrounded by a liquid film suspended in air. The literature on cavitation dates back to Rayleigh. Rayleigh was the first known person to study cavitation by its erosion potential.[11] The properties of cavitation are influenced by the properties of the liquid, such as surface tension, vapor pressure and viscosity.[12] Cavities produce by sound waves can oscillate with the frequency of the sound waves (stable cavitation) or they can violently collapse with sonic pressure forming high-velocity jets of fluid (transient cavitation).[13] Cavities in higher surface tension liquids release higher energy when they collapse. Thus, water shows a greater transient cavitation cleaning action than the organic solvents used in some industrial cleaning methods. When the viscosity of the liquid increases, cavity oscillation and growth are hindered gradually.

The cavity size is mainly determined by the vapor pressure of the liquid. Cavitation action of liquids with high vapor pressure is more rapid than the liquids with low vapor pressure.

In vacuum-generated cavitation, the pressure in a liquid is reduced to slightly below its vapor pressure. Pascal's Law states that all surfaces including interior surfaces see the same pressure.[14] Therefore, cavities can be formed on the exterior surfaces as well as interior surfaces of components with complex geometries. Also, If the pressure is held below the fluid's vapor pressure, cavities will increase in size.[11,15] When the pressure is higher than the vapor pressure of the fluid, the cavity will decrease in size eventually dissolving in the fluid.

Vacuum-generated cavitation is favored by high-energy locations in the fluid. These high-energy areas are adjacent to impurities, defects, and sharp edges on the surface of the components being cleaned. For this reason, cavitation is often observed to begin near scratches on the surface of the component (The reader is encouraged to look for this phenomenon of cavitation near scratches in the bottom of a sauce pan next time they boil water in the stove).

Ultrasonic cavitation cleaning

There are many cleaning methods in the parts cleaning industry and ultrasonic technology is the most commonly used cleaning method due to its low cost and easy maintenance.[16,17] Ultrasonic cleaning technology was developed in the early 1950s. This technology uses sound waves to generate cavitation. Ultrasound is sound with a frequency above 18 kHz that cannot be heard by the human ear.[12] The commonly used frequencies for industrial cleaning are between 20 kHz and 50 kHz.[4,18] There are many

cleaning systems that use frequencies in the 100's of kHz, which have the benefit of reducing the boundary layer and reducing the percentage of potentially damaging transient cavitation. Some systems feature gentle cleaning for semiconductor wafers in the megasonic range with frequencies near 1MHz.[19] If the amplitude of the sound wave is large enough, then cavities form in the liquid inside the cleaning tank. The size of the ultrasonic cavity depends on the ultrasonic frequency. Higher ultrasonic frequencies produce smaller cavities than lower frequencies at equivalent amplitudes.[7]

Damage from transient cavitation

When cavities are created, there is a pressure difference inside (P_i) and outside (P_o) of the cavity that is governed by the Young-Laplace equation.[20] The pressure difference for a spherical cavity is given as follows;

$$P_i - P_o = \frac{2S}{r} \quad (1)$$

where P_i and P_o are inside and outside pressure of the cavity respectively. The S and r represent the surface tension of liquid and cavity radius. Therefore, small cavities generate large pressure differences while large cavities generate small pressure differences.

The ultrasonic cavities grow and collapse with the frequency of the sound. This process is known as acoustic cavitation.[14] As the cavities grow, they experience pressure from the oscillating sound wave. Due to this increasing pressure, the cavity can either shrink (stable cavitation) or implode releasing a microjet of fluid (transient cavitation). These microjets in the turbulent region of the fluid flow randomly in all directions. Those jets that are directed towards the component surface are effective in flushing soil from the surface. At high sonic powers (high amplitude) the violent collapse of transient cavities is

capable of causing damage to the surface of soft metals and coatings. In aqueous solutions, the temperatures and pressures achieved through these collapsing cavities can reach 6000 K and 1000 atm.[13] The mechanism of cavity growth, implosion, and microjet release during the implosion is shown in Figure 3. The sonic shockwaves and streaming liquid microjets can dislodge soil particles and films from the surface of the parts. After the molecular attraction of the soil to the surface of the component is broken, the soil can be flushed away by the laminar and turbulent flow of the cleaning fluid.[12] But, when it comes to the cleaning of components with blind holes and complex geometries, ultrasonic cavitation has difficulty flushing internal volumes effectively as shown in Figure 4.

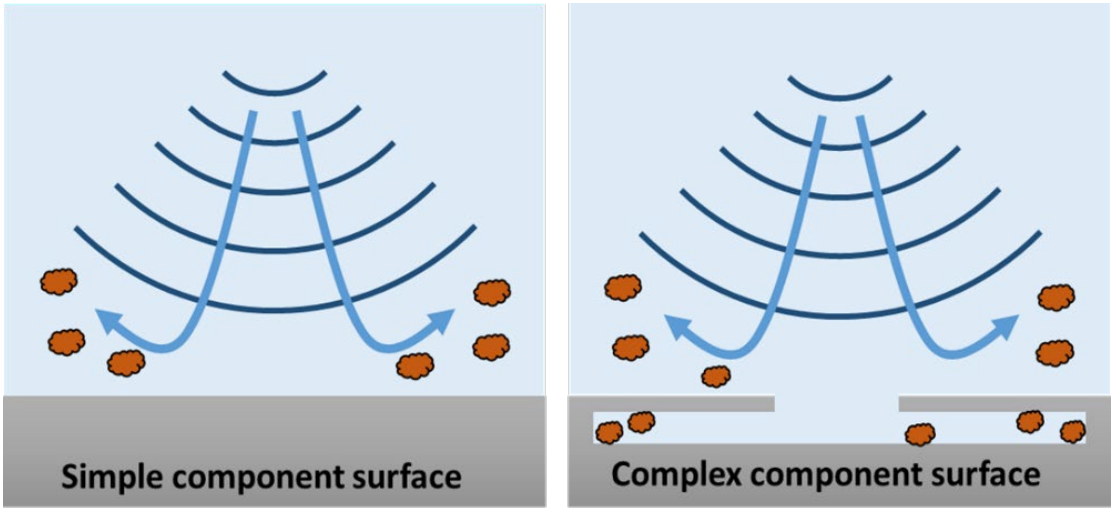

Figure 4. Ultrasonic cleaning with near-surface and interior-surface.

VC and its applications

The cyclic VC cleaning technique is accomplished by reducing the total pressure in a vacuum chamber that contains the cleaning solution and the components to be cleaned. The components are submerged in the cleaning solution. The reduced pressure

results in the formation of cavities at the surface of soiled components. These vacuum-generated cavities form at the solid surface and grow inside the boundary layer.[14] The expanding cavities can push the soil away from the surfaces of the component and disperse the soil into the cleaning fluid as shown in Figure 5. When the vacuum is suddenly removed, the cavities collapse and produce a flushing effect, a density-related inertial effect, and a viscous drag on the surface of the component to be cleaned.[14] On the initial pressure cycle, the cleaning solution is forced into the vacant spaces within the soiled component. Once the soil comes in contact with the cleaning solvent, the soil can enter the cleaning solvent. This washing-mechanical effect can be cycled as often as needed. Therefore, this method is called "cyclic vacuum cavitation cleaning".

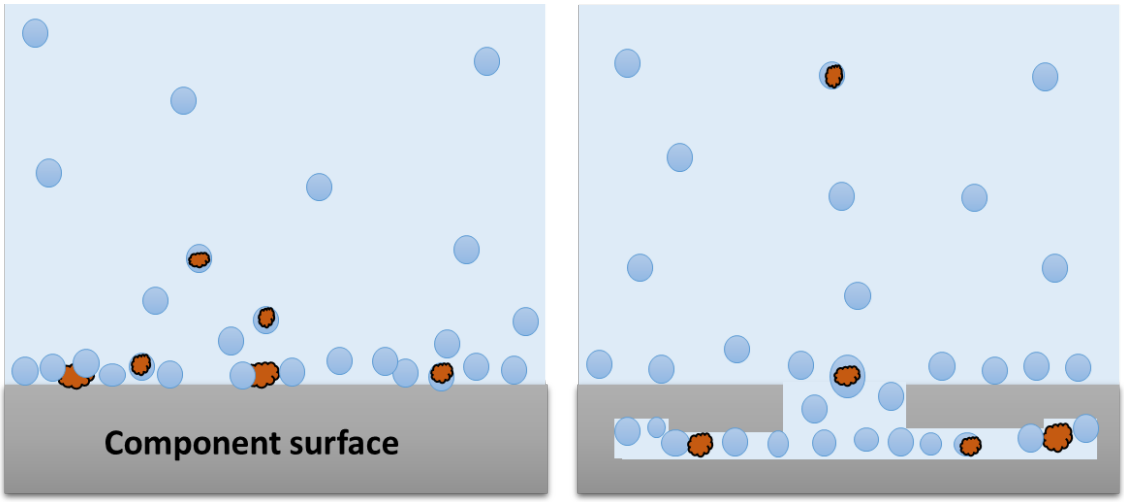

Figure 5. Growth of vacuum generated cavities near-surface and interior-surface.

Tight areas are perfect nucleation points to grow vacuum-generated cavities. The reduced pressure helps to form and grow cavities on these nucleation sites. These growing cavities force fluid out from the component's internal structure. When the vacuum is broken, clean bulk fluid enters the tight spaces of the component. Cycling of

vacuum and ambient pressure removes contaminants and brings fresh cleaning fluid to the surface of the component. Since the vacuum cavitation starts at the component surface, there is no need to break through the boundary layer with potentially damaging microjets. The cavities grow steadily until their size causes them to separate from the surface.[21] Thus, potential damage to the components can be avoided with the cyclic VC cleaning method. Cleaning processes based on VC have been implemented in the semiconductor industry, medical technology, 3D printing, and the optical industry.[21,14]

Capillary tube cleaning using cyclic VC

Capillary tubes act as blind holes. Therefore, they are very hard to clean with traditional cleaning methods. Ultrasonic cavitation has difficulty flushing blind holes efficiently. But, vacuum-generated cavities can grow inside both the cleaning fluid and the capillaries (Figure 6). Since these cavities form on the internal surface of the capillaries, growing cavities can push the soil out of the opening. Breaking the vacuum brings fresh fluid into the capillaries.

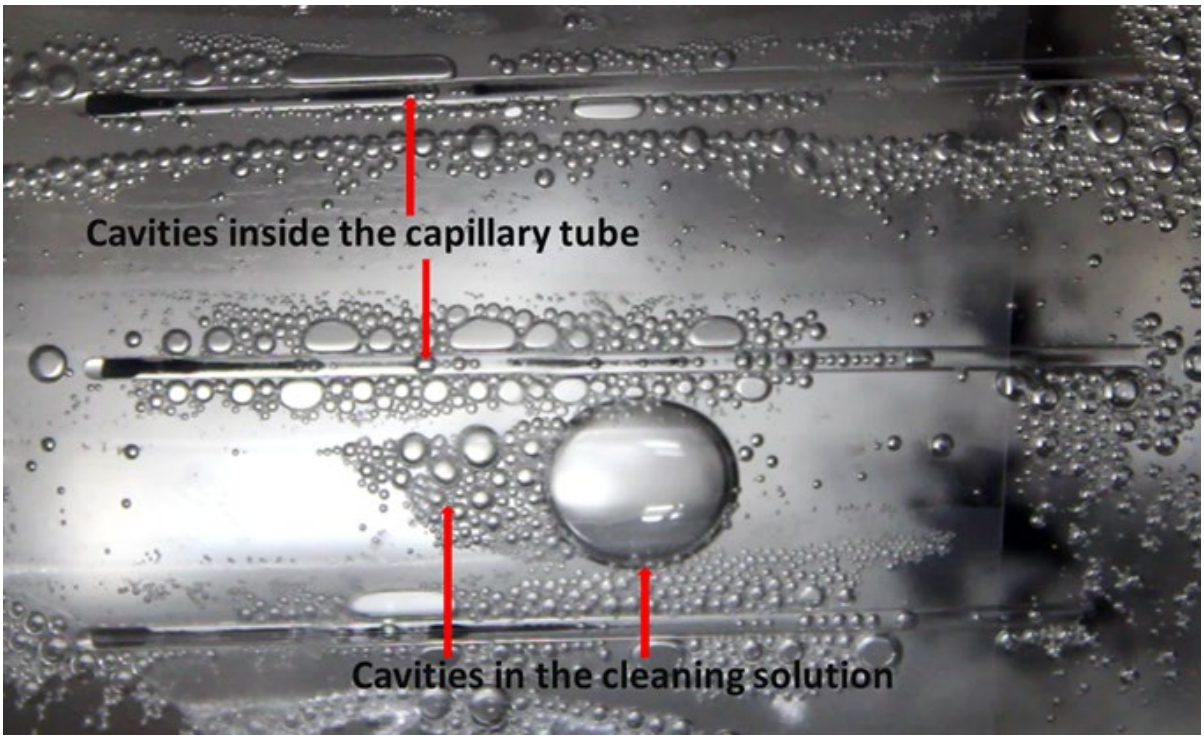

Figure 6. Growing cavities in the vacuum chamber.

Creation of vacuum cavities

The phase diagram of water shows the temperatures and pressures at which the various phases of water can exist and the temperatures and pressures at which the various phases are in equilibrium.[22] The point at which all three lines meet is the triple point. At this point, all three phases of water (ice, liquid, and gas) coexist in equilibrium. At pressures and temperatures above the critical point, a supercritical fluid emerges, which fills the whole container like a gas but has the density of a liquid. If pure water at state A (Figure 7) is depressurized at a constant temperature, liquid water becomes vapor at state C. Also, the state will come to equilibrium at a point such as B. Thus, instead of raising the temperature to the normal boiling point of the water, water can be boiled by reducing the pressure.

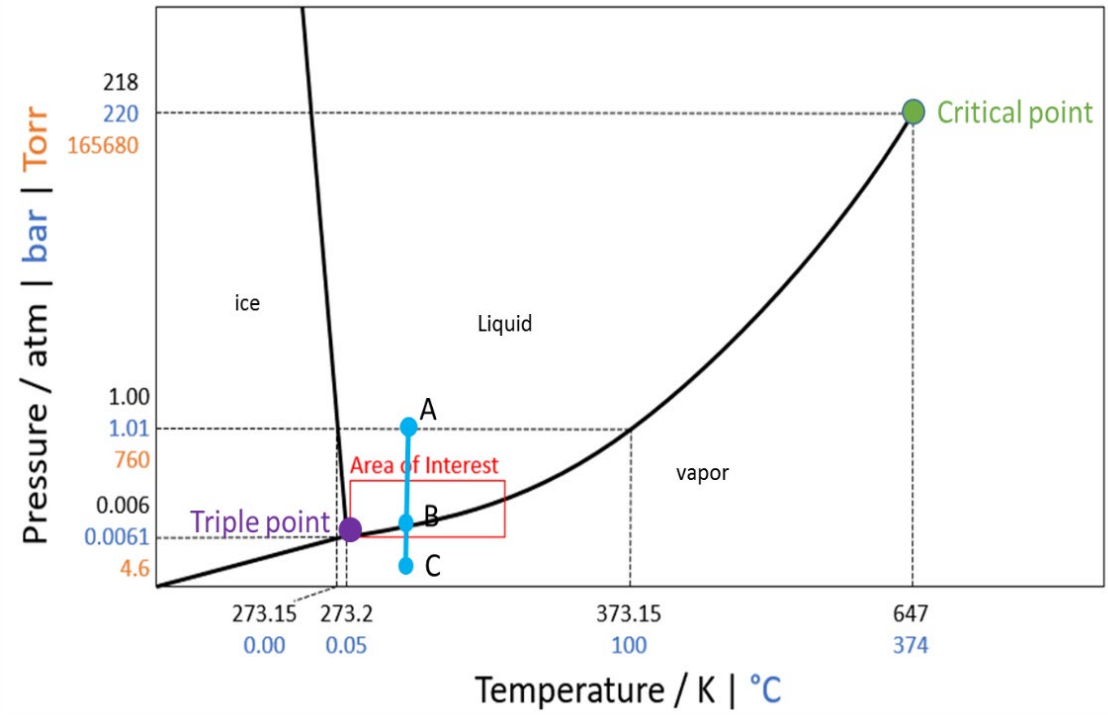

Figure 7. Pressure -temperature diagram of water.

This research is focused on the pressure range between 0.01 and 1.00 bar and temperature between 17.8 and 40 °C. In this region, liquid water starts to form vapor cavities at 0.018 bar at 17.8 °C. Figure 8 shows the vapor pressure of water at different temperatures up to 40 °C. When the temperature of water increases, the kinetic energy of molecules is increased. As the kinetic energy of the molecules increases, the number of molecules transitioning into the vapor phase increases causing an increase in the vapor pressure.

Temperature dependence of cavitation pressure

As described in the previous section, the equilibrium between liquid water and water vapor depends upon the temperature of the system. If the temperature of the water is increased the vapor pressure is increased. This relationship for the two phases is given

by the Clausius-Clapeyron equation. In this case, two phases are water vapor and liquid water. Figure 8 shows how water vapor pressure is increased with the temperature.

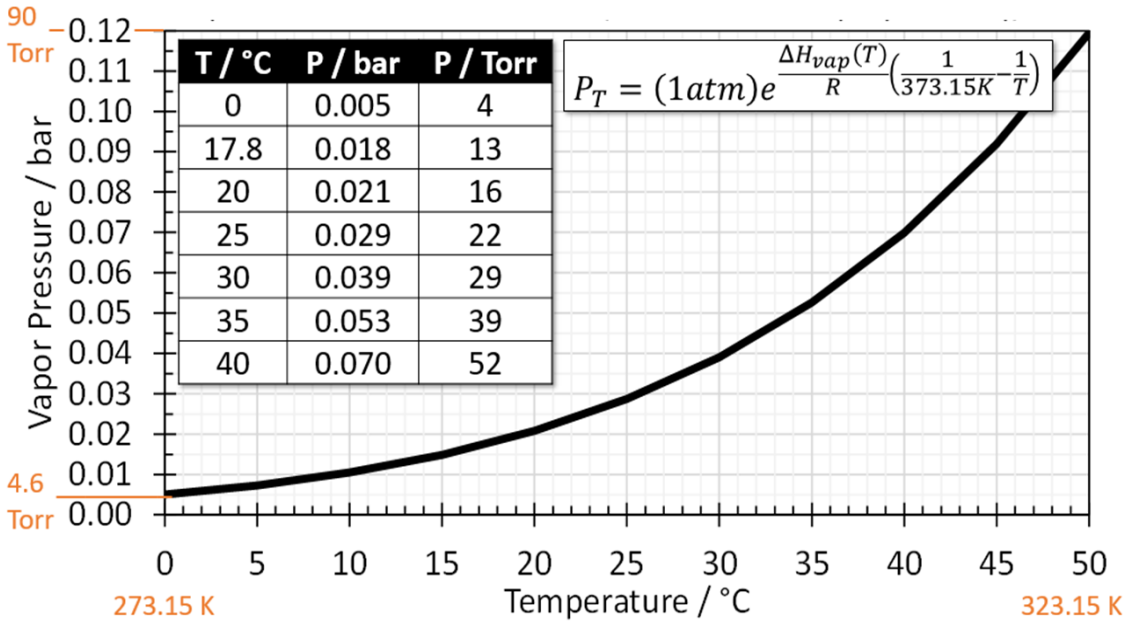

Figure 8. Vapor pressure of water and Clausius-Clapeyron vapor pressure equation.

Vacuum cavitation occurs when the chamber pressure is reduced to the vapor-liquid equilibrium pressure (i.e. the boiling point) at the temperature of the cleaning fluid. This is merely a different way to boil the fluid, reducing the pressure rather than raising the temperature. It should also be noted that temperature changes in the system can alter the cavitation process. Since the vacuum system is pulling the most energetic molecules of liquid vapor out of the chamber, over time, the liquid temperature will drop substantially. A source of heat is often needed to keep the vapor pressure of the liquid high enough to continue cavitation. Also, increasing the temperature reduces the solubility of dissolved gases resulting in better cleaning.[23]

Vapor pressure of 1% SLS in DI water

In this study, sodium lauryl sulfate (SLS) was used as the cleaning solution. Sodium lauryl sulfate is also known as sodium dodecyl sulfate. It is an anionic surfactant commonly used as a cleaning agent in household cleaning products. SLS can be synthetic or naturally produced. It is synthesized by reacting lauryl alcohol from a petroleum or plant source with sulfur trioxide to produce hydrogen lauryl sulfate, which is then neutralized with sodium carbonate to produce SLS.[24] Importantly, it is found that the use of SLS does not cause a risk to consumers or the environment.[24] The vapor pressure of SLS solutions has been studied previously.[25] Peixun Li et al. determined the vapor pressure of the aqueous solutions of SLS ranging from 0% to 10% (mass percent concentration) at different temperatures. Since 1% SLS in DI water was used in the current study, it is important to know the vapor pressure of 1% SLS at different temperatures. This can be also shown using a small calculation. If 18.015 g of water is taken to make a solution of 1% SLS, 0.180 g of SLS is needed. Then, the mole fraction of the solvent can be calculated as follows. The molar mass of SLS is 288.372 g mol^{-1}.

$$X_{SLS} = \frac{moles\ of\ SLS}{moles\ of\ water + moles\ of\ SLS}$$

$$X_{SLS} = \frac{0.00062\ mol}{1\ mol + 0.00062\ mol}$$

$$X_{SLS} = 0.00062$$

$$X_{water} = 1 - 0.00062 = 0.9994$$

Using Raoult's law, the vapor pressure of 1% SLS solution can be calculated at 25 °C.

$$P_{1\%\ SLS\ solution} = X_{water} \times P_{water}$$

$$P_{1\%\ SLS\ solution} = 0.9994 \times 0.029\ bar$$

$$P_{1\%\ SLS\ solution} = 0.029\ bar$$

All these results can be seen in Table 2. From these data, it can be concluded that a 1% SLS solution has essentially the same vapor pressure as pure water.

Table 2. Comparison of vapor pressure values of water and 1% SLS solution at different temperatures.

Temperature / °C	Vapor pressure of water		Vapor pressure of 1% SLS solution / bar	
	bar	Torr	Peixun Li et al data[25]	Raoult's law
17.8	0.018	13	-	0.018
20.0	0.021	16	-	0.021
25.0	0.029	21	0.029	0.029
30.0	0.039	29	0.039	0.039
35.0	0.053	39	0.052	0.053
40.0	0.070	52	0.070	0.070

Surfactant role in cyclic VC

As previously discussed a surfactant reduces the surface tension of the cleaning solution. When the surface tension is reduced, cavitation increases. More cavitation leads to more cleaning and flushing of internal structures. Thus, the addition of a cleaning detergent increases the overall effectiveness of the cyclic VC process. However, at certain concentrations and temperatures, surfactants can create a stable and growing foam layer, which may cause problems with vacuum pumps and with cleaning tanks.

Foaming of surfactants

Foam consists of gas bubbles separated by liquid layers that are normally stabilized by surfactants. Foamability is a measure for the foam-generating power of a surfactant solution while foam stability is a measure for the time foams resist destruction.[26] The foamability of a surfactant depends on the temperature, the type of surfactant, and the concentration. In some cleaning processes, foams can be used to remove soil from the components. For example, in the cleaning of radioactive vessels, a good foaming agent is applied.[27] In cyclic VC cleaning, foam is not desired as it can enter the vacuum system potentially degrading its effectiveness. Oil-based vacuum systems do not perform well with substantial water contamination. Inexpensive water aspiration vacuum systems are not affected by water vapor, surfactant, or foam absorption and are therefore preferred.

Methods for monitoring surface cleanliness

Reliable methods are needed to determine the cleanliness of components. Normally, quantitative methods are preferred over qualitative methods as they can be easily applied for statistical evaluation. In many industries, cleaning is evaluated by visual inspection. Qualitative methods of evaluating cleanliness like visual inspection vary from place to place mainly due to the variability in the analyst. Photographic and video analysis can reduce or correct human errors in the results. Photographs of parts before and after cleaning can be analyzed in image software to calculate quantitative cleanliness values. Advanced microscopy techniques can be used such as visible, infrared, Raman, transmission electron microscopy (TEM), scanning electron microscopy-energy dispersive X-ray spectroscopy (SEM-EDS), and atomic force

microscopy (AFM). There is a trade-off between magnification and field of view, and these techniques require viewing access or direct access to the surface that was cleaned.[16] It is difficult to evaluate cleanliness in 50 mm × 1 mm capillary tubes using those techniques. Therefore, in this study, gravimetric analysis was used to calculate the percentage removal (%R) of soil from the tubes.

Gravimetric analysis is a common method for measuring cleanliness because it is relatively inexpensive requiring only an analytical balance. It can detect soils on component surfaces down to the $\mu g/cm^2$ level and is a direct and rapid analytical method. However, it is not as sensitive as high-performance liquid chromatography (HPLC) on extracted soils. Expensive instrumental analytical techniques like HPLC require a soil that is soluble in the mobile phase, which is a problem with a sooty soil like our carbon black sample.

CHAPTER II

Material and Methods

Chemicals and other materials

The surfactants examined in this study are shown in Table 3.

Table 3. List of surfactants used in this study.

Name	Form	Supplier
Sodium lauryl sulfate (SLS)	Powder	
Alconox	Powder	Alconox
Detergent 8 solution	Liquid	Alconox
Hexadecyltrimethylammonium bromide (CTAB)	Powder	

Other materials used in this study include a vacuum chamber (Figure 9), rotary vane vacuum pump (Welch 8907A), water aspirator vacuum pump (Eyela A-1000S), temperature probe (P4015 digital thermometer), high-resolution camera (Canon Rebel T6i), 45 kHz ultrasonic bath (Crest ultrasonics, P500D), oven, USB microscope (Celestron, 44308), 4-place analytical balance (Ohaus Adventurer Pro AV64), 5-place analytical balance (KERN ABT120-5DM), and a stirring hot plate (Fisher Scientific Isotemp). Carbon black powder (Jacquard, PearlEx 640, particle size 10 to 60 microns) was used as the model soil in all the experiments.

Gravimetric analysis

In the present study, before the cleaning process began, the empty weight of individual capillary tubes was measured. The capillary tubes were filled with carbon black powder soil and the weight of soil in tubes was measured. The contaminated

samples were cleaned. The weight of the cleaned samples was measured and recorded. All of the weights mentioned above were measured in grams (g). The soil removal percentage was calculated based on the general equation below: If the mass of the carbon black powder soil for before and after cleaning scenarios is represented as M_{CB}^{before} and M_{CB}^{after} respectively, %R can be calculated using Eq. 2.

$$\%R = \frac{M_{CB}^{before} - M_{CB}^{after}}{M_{CB}^{before}} 100\ \% \quad (2)$$

Structural & Operational Information about cyclic VC

One of the main purposes of this project was to construct a VC apparatus for use for blind hole cleaning. The VC apparatus includes a vacuum chamber, a vacuum pump, a hose to connect the chamber to the pump, valves to control the vacuum, a vacuum pressure gauge to measure the pressure inside the chamber, and a temperature probe to measure the temperature inside the vacuum chamber. The components to be cleaned are submerged in a bath of cleaning liquid. The vacuum pump applies a negative gauge pressure to the chamber to remove air. Pulling air from the vacuum chamber can produce cavities on the component surface and inside blind holes and pores. Purging the vacuum with room air collapse the cavities bringing fresh cleaning fluid to the component surface and inside blind holes and pores. This process is called one vacuum cycle. Temperature can be controlled using a hot plate. Visual monitoring of the chamber interior is made possible through a clear plexiglass lid (Figure 9).

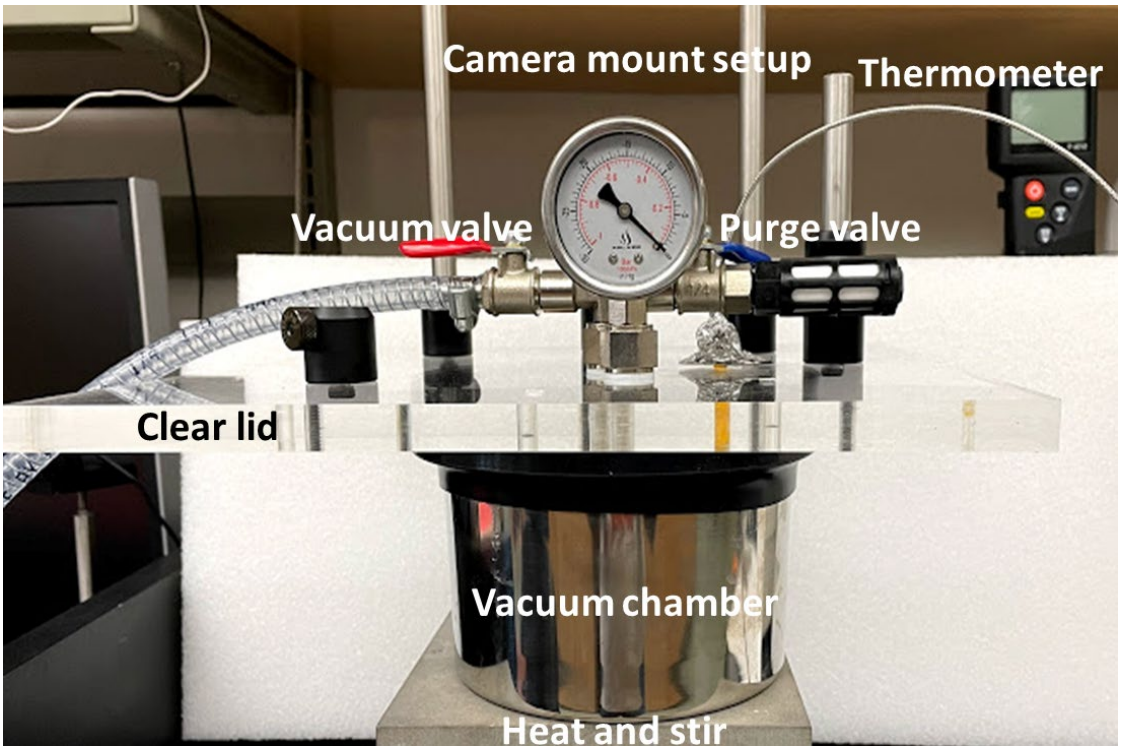

Figure 9. VC apparatus.

Figure 9 illustrates the VC system. The blue purge valve is coupled to the chamber to release the chamber pressure. The red vacuum valve is used to pull the vacuum. The vacuum chamber can be kept on a hot plate to heat the cleaning liquid to a required temperature. The cyclic vacuum cavitation process can be performed by opening and closing the red and blue valves. The vacuum chamber can be heated or cooled to maintain a constant temperature of the liquid at the proper pressure. The general operational procedure of cyclic VC cleaning can be as follows;

1. Set the camera, hot plate, and digital thermometer.
2. Tape the capillary samples inside the cleaning system.
3. Submerge the samples in the cleaning liquid.

4. Close the purge valve and open the vacuum valve to pull the vacuum. It is important to check the pressure gauge to confirm the pressure is reduced. To achieve 0.01 bar pressure normally takes 20 seconds.
5. Observe cavity formation inside the cleaning liquid and capillary tubes.
6. When new cavities cease forming in the capillary tubes, close the vacuum valve and open the purge valve to purge the vacuum with room air. This will bring the pressure inside the chamber to ambient pressure which is close to 1 bar atmospheric pressure.
7. Repeat steps 4,5 and 6 to cycle the VC cleaning process.

Surfactant solutions were replaced with fresh samples after each trial. The vacuum chamber was cleaned routinely before and after each experiment with tap water and deionized water. Only a soft sponge and absorbent paper tissues were used to contact the stainless steel chamber. The temperature inside the chamber was maintained as a constant throughout the experiment. After using the rotary vane vacuum pump for the preliminary experiments, it was changed to the water aspirator vacuum pump due to its advantages over the rotary vane vacuum pump.

Recirculating water aspirator pump

Using a water aspirator pump is an inexpensive way to generate a vacuum. The ultimate pressure of the aspirator pump is determined mainly by the vapor pressure of the liquid in the aspirator tank.[28] Since ice water has lower vapor pressure than water at higher temperatures, the aspirator tank was filled with ice water before the experiment. The aspirator tank is not insulated. So, as the water in the tank recirculates, it warms over time. Also, vapors that are created in the chamber will come to the aspirator tank and will

be diluted by water. The aspirator pump is useful in this study since no precautions need to be taken against the entrance of vapor or foam into the aspirator where they are rapidly diluted with water.[29] Figure 10 shows the water aspirator pump model. In this model, high-velocity water is going through the nozzle into the throat creating a pressure difference due to the venturi effect. Then the air will be sucked from the vacuum chamber to equalize the pressure. This process will create a vacuum inside the chamber.

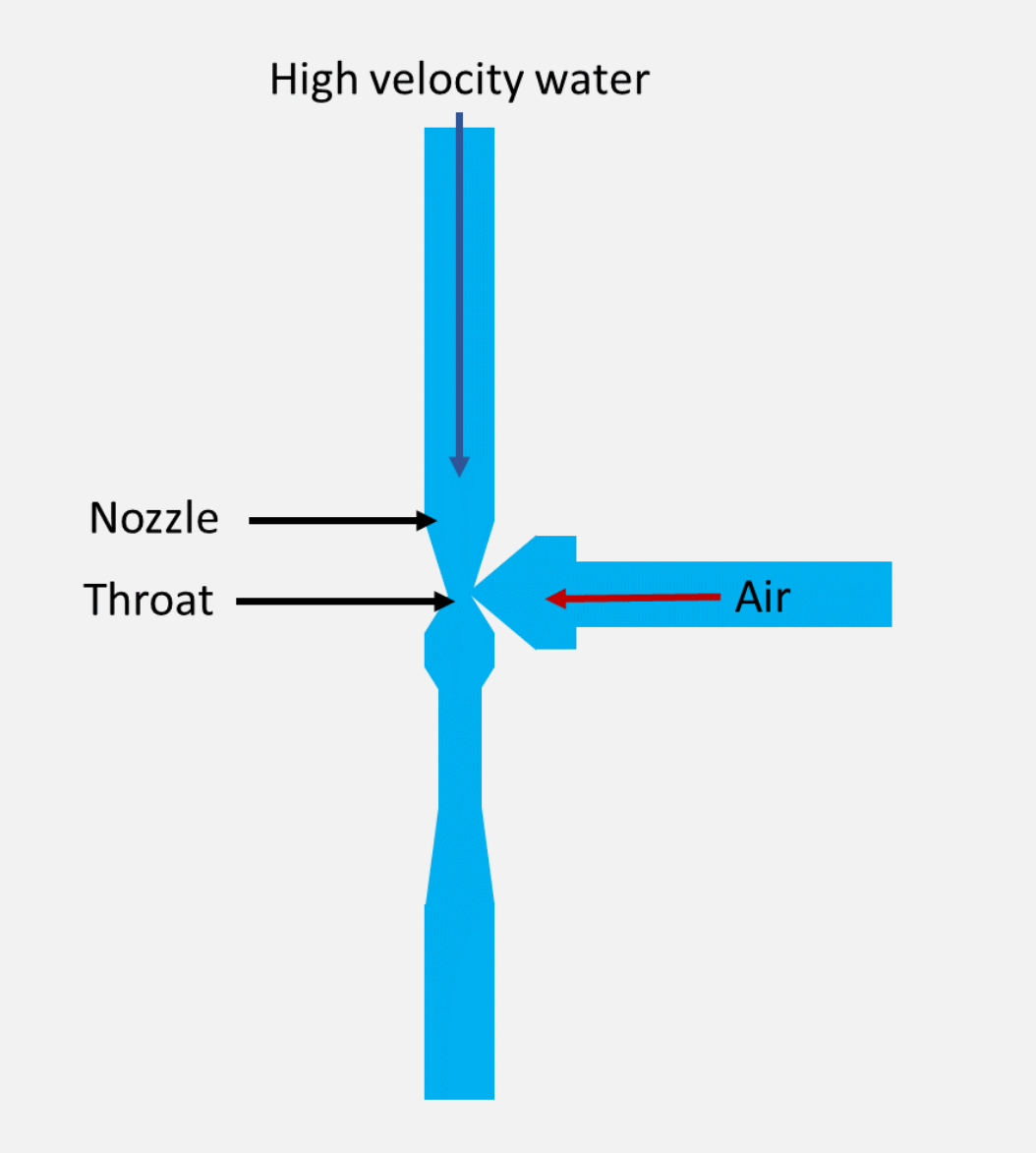

Figure 10. Water aspirator pump model.

Pressure gauge

The pressure gauge measures the pressure inside the vacuum chamber relative to atmospheric pressure. Gauge pressure is positive for pressures above atmospheric pressure and negative for pressures below it as shown in Figure 11. Therefore, the absolute pressure can be written as follows;

$$P_{abs} = P_g + P_{atm} \tag{3}$$

where P_{abs}, P_g and P_{atm} are absolute pressure, gauge pressure and ambient atmospheric pressure respectively. The lowest and highest pressures that can be achieved using this pressure gauge are 0 bar and 1 bar respectively.

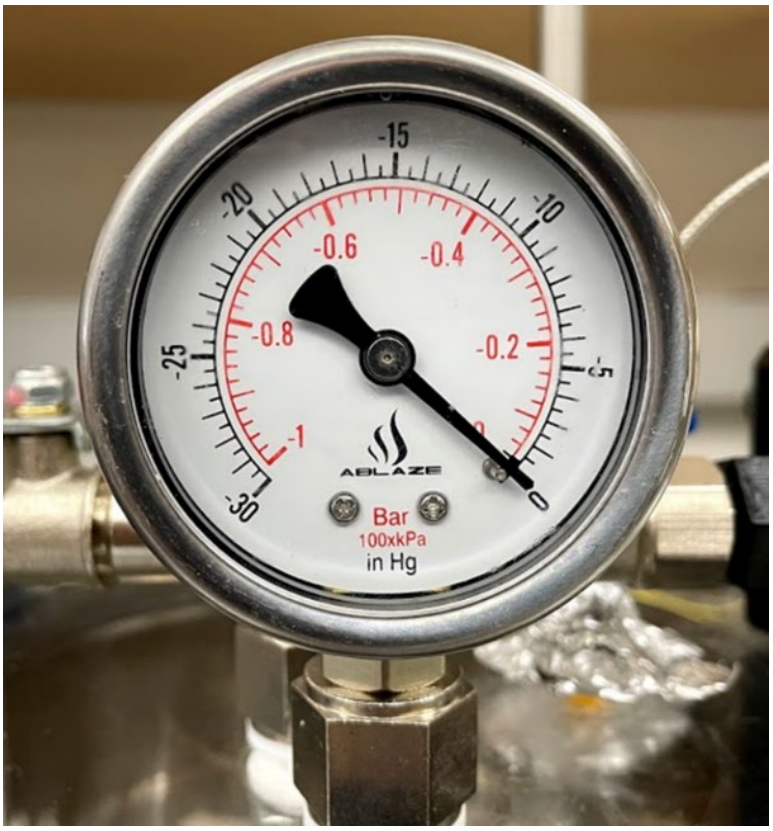

Figure 11. Pressure gauge showing 0 inches of mercury gauge pressure (P_g).

Observation of cavitation

The observation of cavities in the capillaries is a useful step in cyclic VC cleaning. The Canon Rebel T6i camera and Celestron microscope were used to record the cleaning process. Thick plexiglass windows were made to get a better view of the cavities inside the chamber. They were made using a miter saw and a mill (Figure 12).

Figure 12. Making plexiglass windows using the miter saw (left) and mill (right).

One rectangular window and two prisms were made for experimental purposes. Two prisms were taped together to see the reflection of vertical capillary tubes from the top surface of the plexiglass window (Figure 13). The lower of the two prisms served no optical purpose. It was merely used as a base that ensured the top prism could reflect the horizontal view up to the camera.

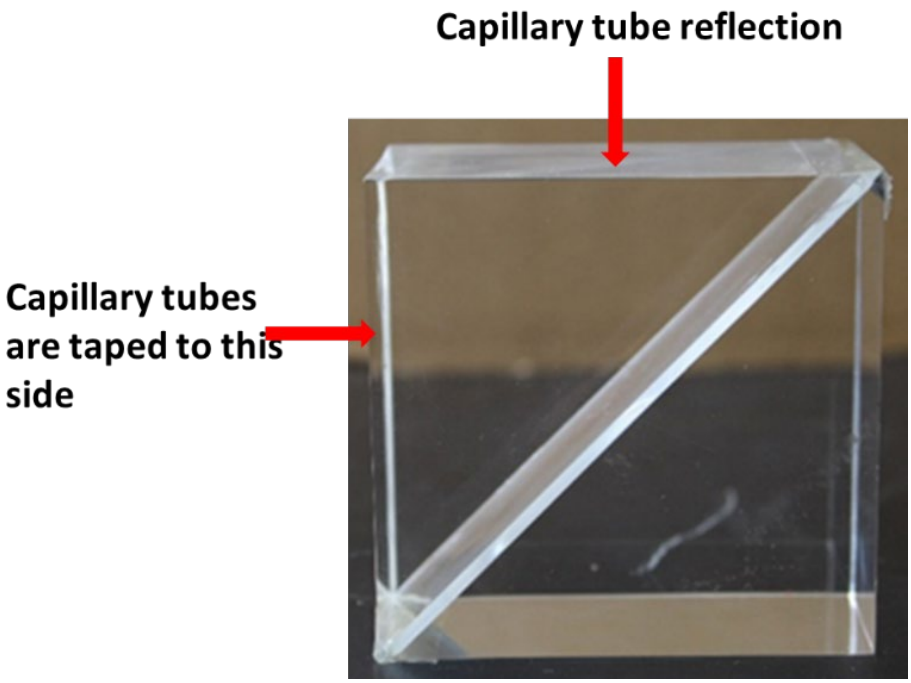

Figure 13. Observation of vertical capillary tubes using two prism plexiglass windows.

Sample Preparation

The outer and inner diameters of the capillary tubes were measured using the Celestron microscope and analyzed using ImageJ software (Figure 14). The dimensions were measured using a caliper micrometer. The outer diameter, inner diameter, inner length, and outer length of capillary tubes were 1.46, 1.12, 49.00, and 50.00 mm respectively.

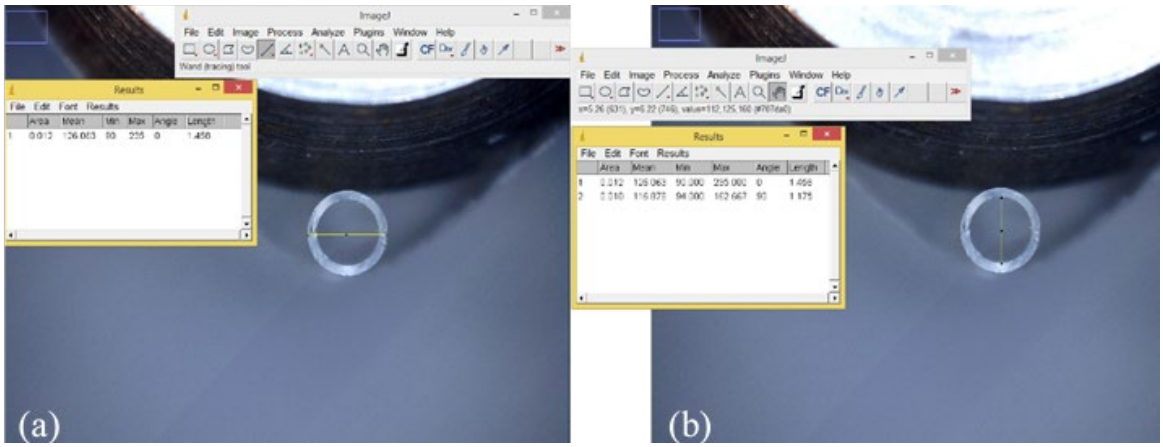

Figure 14. ImageJ analysis of (a) outer and (b) inner diameter measurements of the capillary tube.

All the surfactant solutions were prepared by dissolving the surfactant (1% SLS in 99% DI water) in deionized water. Carbon black powder was used as the model soil in all the experiments because it is not soluble in the cleaning solutions, is easy to track photographically, and will be difficult to remove from blind holes. The amount of material before and after the cleaning was measured using the balance. Both dry and wet capillary tubes were prepared for cleaning analysis. Capillary tubes were soiled with carbon black powder up to 15.00 mm in length by tapping. Wet capillary tubes for ultrasonic experiments were prepared using one vacuum cycle in the vacuum chamber to fill them with the cleaning solution. These prepared wet capillaries are shown in Figure 15. After the cleaning treatment, the cleaned capillaries were dried using the oven at 120 °C for 1 hour.

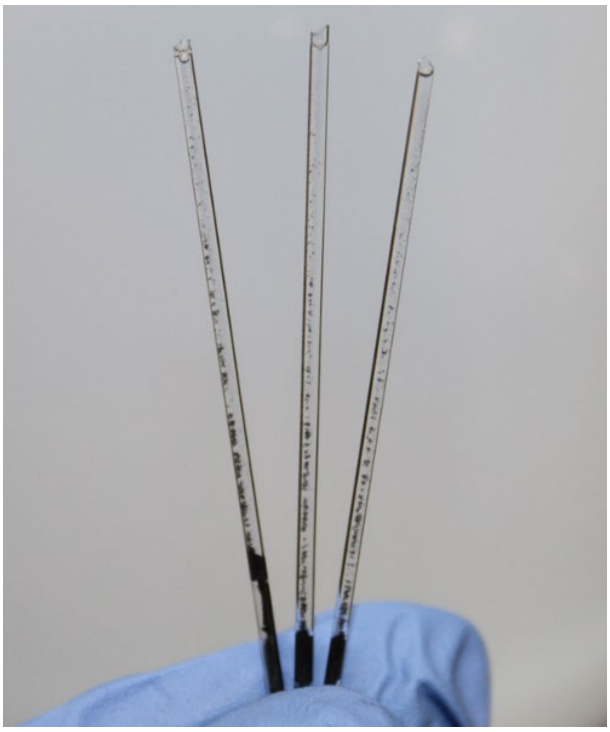

Figure 15. Soiled wet capillary tubes.

Contamination level of carbon black powder in capillary tubes

The inner contamination levels of capillary tubes were calculated using average soil mass before and after the cleaning process and the inner surface area of the capillary tube. Considering the capillary tube as a cylinder, the inner surface area (A) can be calculated using Eq. 4. The inner volume of the capillary is mostly cylindrical with a half-spherical glass bead at the bottom.

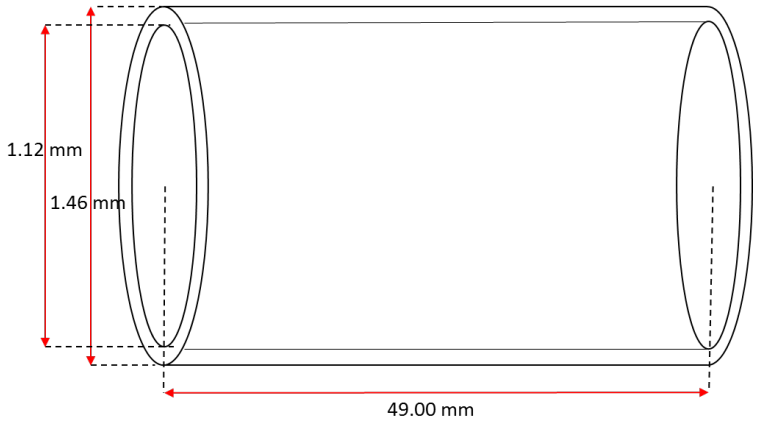

Figure 16. Inner, outer diameters and the inner tube length of the capillary tube.

$$A = \pi r^2 + 2\pi rh \tag{4}$$

According to the inner diameter and height of the capillary tube, the surface area can be calculated as follows;

$$A = \left(\pi \left(\frac{1.12\ mm}{2}\right)^2\right) + \left(2\pi \left(\frac{1.12\ mm}{2}\right) 49.00\ mm\right)$$

$$= 1.73\ \times 10^2\ mm^2$$

$$= 1.73\ cm^2$$

The limit of detection (LOD) value of the balance

The detection limit of the balance is defined as the minimum mass that can be detected at a known confidence level. Also, it can be expressed as the mass of the analyte at which the signal-to-noise ratio is equal to 3. Below this limit, detection of the signal becomes less statistically confident. The uncertainty of the KERN ABT120-5DM analytical balance is ± 0.05 mg. Since carbon black powder is measured before and after the cleaning process, the uncertainty for each experiment from the balance can be calculated as follows;

$$S_{result} = \sqrt{S_{before}^2 + S_{after}^2}$$

$$= \sqrt{(0.05\ mg)^2 + (0.05\ mg)^2}$$

$$= 0.07\ mg$$

Then the LOD of the balance becomes 0.21 mg (3 ×0.07 mg). The LOD contamination level for the balance can be calculated by dividing the LOD of the balance by the inner surface area of the capillary tube. Therefore, the contaminated levels below 121 μg cm^{-2} can not be detected confidently using this analytical balance.

In this study, many of the results showed contamination levels below the LOD of the balance. These were indicative of clean samples, but also indicative of our relatively insensitive gravimetric cleanliness monitoring method. For example, contamination levels for many precision cleaning industries are in the ng cm^{-2} range.[30]

Screening of surfactant foaming properties using the cyclic VC method

The effect of the temperature on foam formation in cyclic VC was studied using four surfactant solutions with 1% mass percent concentration. They are sodium lauryl sulfate (SLS), hexadecyltrimethylammonium bromide (CTAB), detergent 8 solution, and alconox. The vacuum chamber was filled with 400 mL surfactant solutions and exposed to a vacuum of 0.01 bar for 1 minute at 18°C, 22°C, and 28°C temperatures. Foam formation was recorded with the camera.

The number of vacuum cycles

After setting up the apparatus, soiled capillaries were taped to the plexiglass window horizontally and submerged in the cleaning solution. The vacuum was pulled to 0.01 bar and purged with the room air to collapse the cavities and bring fresh fluid into the capillary. This is considered as one vacuum cycle. After the cleaning process,

capillaries were dried in an oven at 120° C for 1 hour, and %R was calculated using Eq. 2.

Comparison of cyclic VC cleaning to ultrasonic cleaning

A conventional cleaning method, ultrasonic cavitation (UC) cleaning with SLS solution was used to clean the same soiled capillary tube samples to compare against the cyclic VC cleaning method. The cleaning agent was pre-mixed with deionized water at a mass ratio of 1: 99 (cleaning agent: water). The contaminated tubes were submerged in the cleaning solution and then exposed to 45 kHz ultrasonic cleaning for 5 minutes. The leftover solution was removed and the parts were dried in an oven at 120° C for 1 hour to remove excess water droplets and the percentage removal calculation was conducted as prescribed in Eq. 2.

To further analyze the effect of capillary tube orientations in these two cleaning processes, two sets of soiled capillary tubes were taped horizontally to the rectangular plexiglass window, submerged in the1% SLS/DI solution, and cleaned with VC and UC cleaning. Another two sets of soiled capillaries were taped vertically to the plexiglass prism window, submerged in 1% SLS/DI solution, and cleaned with the cyclic VC and UC cleaning. An additional two sets of soiled wet capillaries were taped horizontally and vertically to the rectangular plexiglass window, submerged in the1% SLS/DI solution, and cleaned with UC cleaning. After each cleaning process, the cleaned capillaries were dried in the oven at 120° C for 1 hour. The %R was calculated based on Eq. 2. The temperature inside the vacuum chamber and ultrasonic bath were maintained at room temperature.

Cyclic VC cleaning at different temperatures

The vacuum chamber was filled with 1% SLS/DI solution and the temperature of the solution was varied from 25, 30, 35, and 40 °C using the hot plate. Soiled capillaries were taped horizontally to the plexiglass window and submerged in the heated 1% SLS/DI solution and cleaned with 15 cycles. When the temperature was increased in the vacuum chamber, the steam formation interfered with the view of cavity formation in the capillary tubes. Therefore, plexiglass windows were taped to the lid of the chamber during these experiments (Figure 17). After each cleaning process, the cleaned capillaries were dried in the oven at 120° C for 1 hour. The %R was calculated based on Eq. 2.

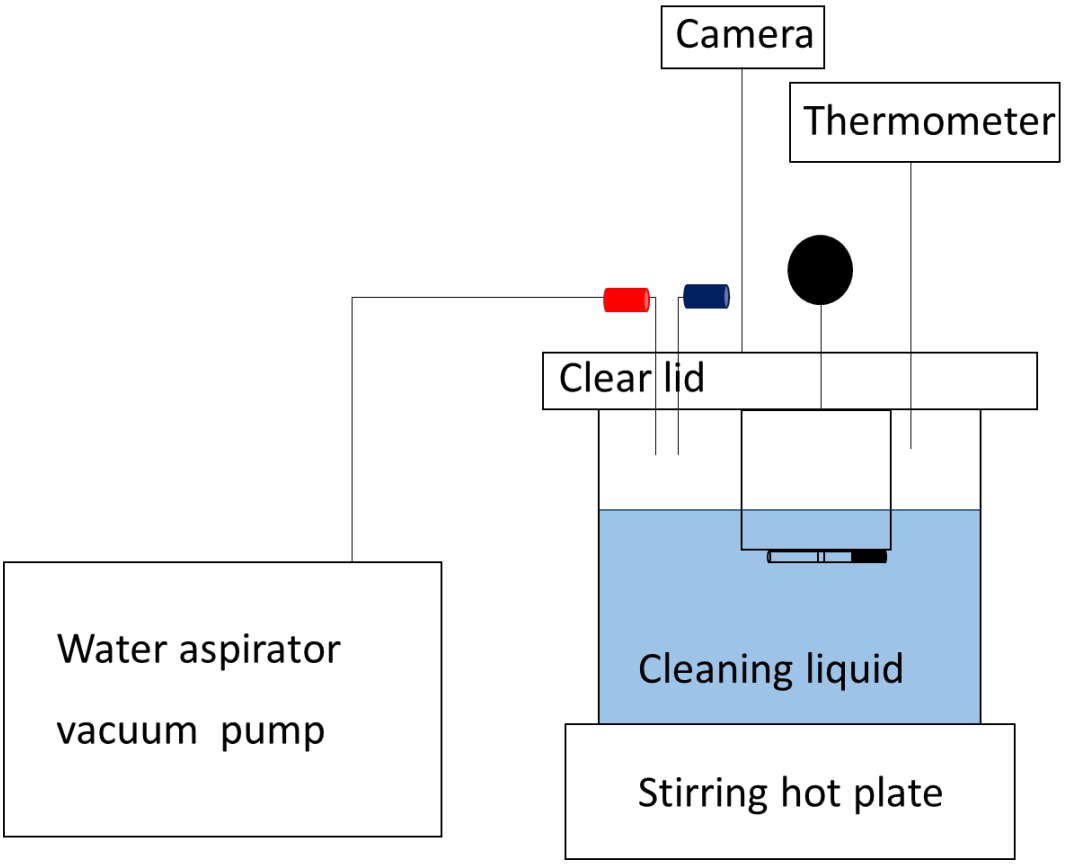

Figure 17. Experimental setup for temperature experiments with horizontal capillary tubes.

Tape residue confirmation study of the capillary tubes

Note that the capillary tubes were taped to the plexiglass window when the experiments were performed. Therefore, residues from the tape might have been attached to the capillary tube after the cleaning process and these residues might influence the data and/or the cleaning process. An experiment was done to determine this possibility. Ten empty capillary tubes were taped horizontally and vertically to the rectangular plexiglass window and cleaned with 1%SLS/DI solution in the vacuum chamber using 15 cycles. After the cleaning process, they were dried in an oven for 1 hour. Then before and after masses of capillary tubes were compared.

Statistical analysis

Two-sample t-test analysis and multi-sample t-test analysis were performed using Minitab 17. The p-values from these statistical tests are the probabilities that the null hypothesis is true. As probabilities, p-values range from 0 to 1. In this study, 0.05 is used as the α value. Therefore, if the p-value of a test statistic is less than 0.05, the null hypothesis is rejected. For example, consider a two-sample t-test to test the difference between the mean percent removals of two cleaning methods. In this case, the null hypothesis states that the two sample means are equal while the alternative hypothesis states that they are not equal. A p-value below 0.05 suggests that the population means are different with at least 95% confidence ($1-\alpha$).

Final confirmation study of cyclic VC cleaning

The final confirmation study used all the successful parameters from the previous studies to optimize the cleaning efficiencies in the cyclic VC cleaning method and will be discussed in the results.

CHAPTER III

Results and Discussion

This chapter contains data and discussions that were collected using the methods outlined in Chapter II**.**

Cavity observation and cyclic VC cleaning

The observed results showed that cavities were formed after the pressure reduction in the vacuum chamber. At first, the size of cavities increases as the pressure decreases in the chamber. When the pressure starts to return to atmospheric pressure, cavities collapse drawing cleaning fluid into the spaces the cavities occupied. The growing cavities pushed substantial amounts of carbon black powder from the capillary tubes with each vacuum cycle. Figure 18 shows images of cavity growth and the cleaning process. The observed times of each image (10s and 12s) are displayed on the image relative to the starting time of the pressure reduction. Large cavities were formed in the solution while small cavities were formed inside of the capillary tube. These small cavities were growing inside the capillary tube and were responsible for the internal cleaning. When the cavities are coming out from the capillaries, they were also able to push the soil away from the capillary tubes. This indicates that the cleaning is related to the movement of cavities.

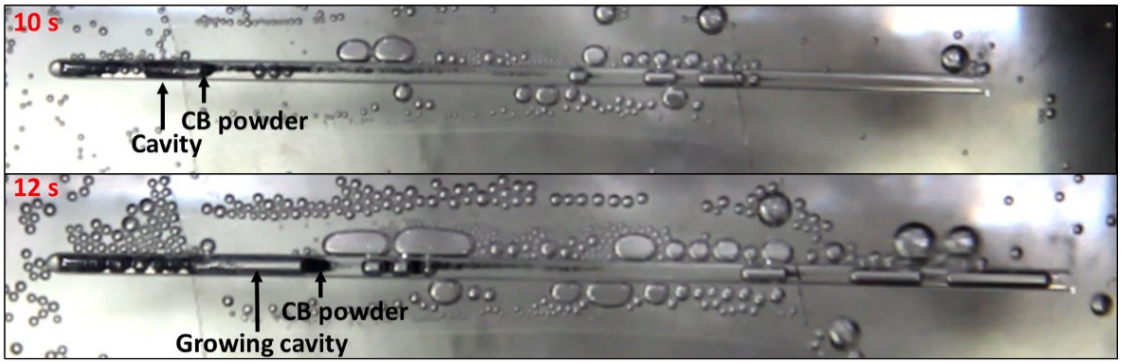

Figure 18. Cleaning process by cavitation effect.

Foam behavior of surfactants

The effect of the temperature on foaming was studied using four 1% surfactant solutions in DI water using the vacuum chamber. Images of foams and cavities for the temperature data with different surfactant solutions are presented in Figure 19. It can be noted that the foaming is decreased with increasing temperature. Foaming is not desired in cyclic VC cleaning, as it can enter the vacuum system potentially degrading the effectiveness of the vacuum pump. Therefore, the increasing temperature in the vacuum chamber to reduce the foaming in the cleaning solution is advantageous. According to the results, 1% detergent 8 in DI water gave the lowest foaming effect when the pressure was reduced, but it was not used because of its proprietary composition. Since the cavities were not observable through the white CTAB solution, it was also not used as a cleaning solution. Since alconox gave more foaming than SLS at higher temperatures, the best option was 1% SLS in DI water for cleaning purposes. Also, SLS is an inexpensive surfactant and can be easily purchased. According to the previous studies and Raoult's law calculations, 1% SLS in 99% DI water has the same vapor pressure as water.

Considering all these results, 1% SLS/DI solution was used as the cleaning solution in the current study.

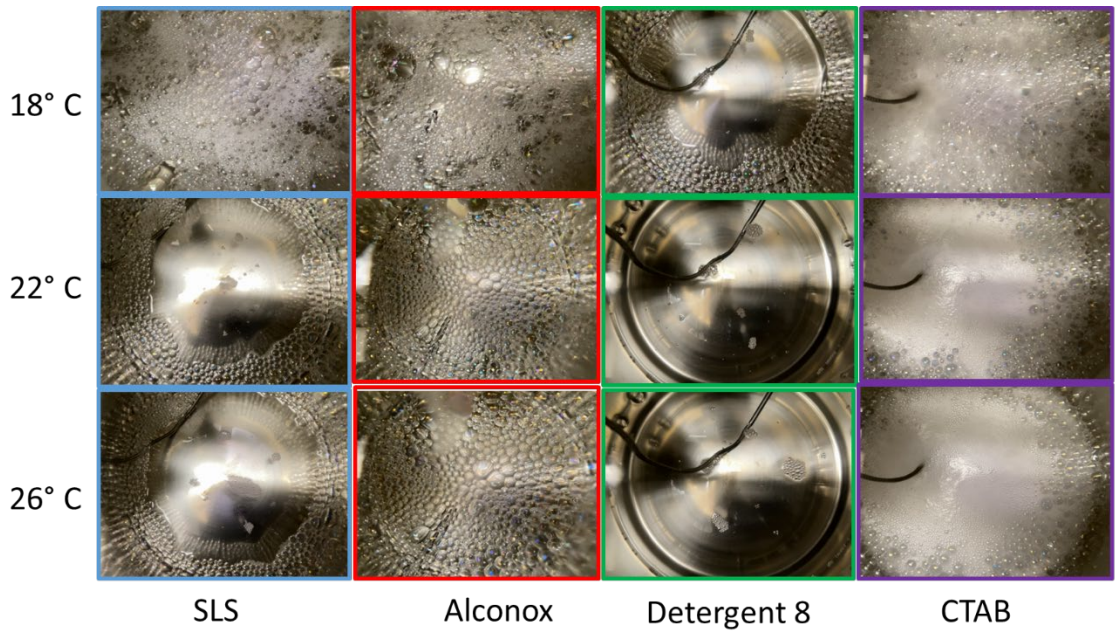

Figure 19. Images of foam and cavity formation of 1% surfactant solutions in DI water at different temperatures in the vacuum chamber.

Cyclic VC cleaning with DI water and 1% SLS/DI water

As the first mode of cleaning experiments, DI water and 1% SLS/DI were used to remove CB powder from capillary tubes. The carbon black powder was used as the model soil because it is similar to sooty carbon contamination from overheated cooling fluids. Sooty carbon soil is common in many industries.[31] Soils of all types become hard to clean when trapped in tight spaces of components with complex geometries and in blind holes, which are similar to our capillary test pieces. After the capillaries were treated with cyclic VC cleaning, the percentage removal of carbon black powder from capillaries was measured. In Figure 20, the cleaning efficiencies (%R) between DI water and 1% SLS/DI solution are compared at room temperature. In this experiment, all the

capillaries were placed horizontally in the vacuum chamber. Significant amounts of carbon black powder remained in the capillary tubes after the fifteen cycles of VC treatment. It can be seen that 82 ± 7% of the carbon black powder is removed from the capillary tubes after 15 cycles using 1% SLS/DI solution, indicating that the cleaning efficiency of using 1% SLS/DI solution is higher than treatment with DI water alone at room temperature.

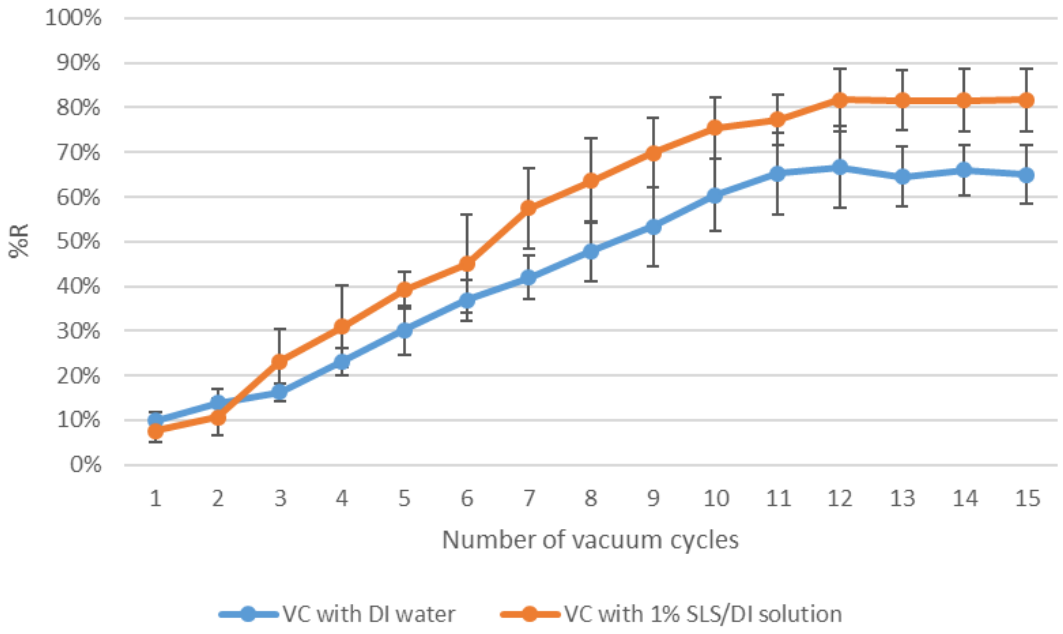

Figure 20. Percentage removals of CB powder with the number of vacuum cycles at room temperature.

This is understandable because the removal of carbon black powder from the capillaries by 1% SLS/DI solution is aided by the low surface tension of the surfactant solution. However, most of the cleaning was taking place in the initial twelve cycles, which results in 82 ± 7% and 67 ± 7% of the carbon black powder being removed with 1% SLS/DI solution and DI water, respectively. In this case, the remaining carbon black

powder was strongly attached to the bottom of the capillary tube. It was difficult to get the cleaning solution to fully wet the very bottom of the tube making the soil in the bottom and resistant to removal. This suggests that it is necessary to wet the internal surface of the bottom of the capillary tube with the cleaning solution to get better removal. As a solution for this problem, the temperature of the cleaning solution was increased and the results of temperature experiments were detailed on page 48.

Comparison of cyclic VC cleaning to ultrasonic cleaning

To compare cyclic VC cleaning to ultrasonic cleaning, six cleaning treatments were used. Table 4 displays the percentage removals of carbon black powder with each cleaning treatment. 1% SLS/DI solution was used as the cleaning solution at room temperature. Capillary tubes were placed horizontally and vertically in VC and cleaned with fifteen cycles of cavity formation and removal. A 45 kHz bath was used to clean dry and wet capillary tubes using the ultrasonic treatment for 5 minutes. In this case, it was apparent that the highest % R was observed when capillaries were placed horizontally in VC with 82 ± 7% removal. On the other hand, the highest percentage removal was observed for UC cleaning was the vertical dry capillaries with 56 ± 12% removal. Following each treatment in VC, the amount of CB powder removed in the capillaries is higher than all the treatment methods in ultrasonic cleaning because UC was unable to flush the carbon black soil from the internal volume of the capillary tubes.

Table 4. Comparison of cleaning ability (%R) of vacuum cavitation (VC), wet/dry ultrasonic cavitation (UC), and vertical/horizontal sample orientation at room temperature.

Cleaning method	Number of cycles or frequency / time	Cleaning solution	% R
Horizontal wet VC	15 cycles / 5 minutes	1% SLS/DI water	82 ± 7
		DI water	67 ± 7
Vertical wet VC	15 cycles / 5 minutes	1% SLS/DI water	65 ± 5
		DI water	60 ± 14
Horizontal dry UC	45 kHz / 5 minutes	1% SLS/DI water	23 ± 11
		DI water	11 ± 4
Horizontal wet UC	45 kHz / 5 minutes	1% SLS/DI water	10 ± 3
		DI water	9 ± 2
Vertical dry UC	45 kHz / 5 minutes	1% SLS/DI water	56 ± 12
		DI water	53 ± 3
Vertical wet UC	45 kHz / 5 minutes	1% SLS/DI water	27 ± 2
		DI water	19 ± 5

In horizontal dry ultrasonic cleaning, only the vibration of carbon black powder was seen in the capillary tube. This observation is shown in Figure 21. Due to this, very little soil is flushed out from the capillary tubes whereas, in cyclic VC cleaning, the flushing effect of the growing cavities was observed as in Figure 18. In horizontal wet ultrasonic cleaning, carbon black powder was stuck to the bottom of the capillary tube and was not be able to come out from the tubes. Therefore, it gave the lowest percentage removal compared to the other methods.

Figure 21. Effect of ultrasonic vibration in horizontal dry capillary tube cleaning.

When vertical dry capillaries were used in ultrasonic cleaning, carbon black powder was stuck in the tip of the capillary tubes and was not able to come out from the tubes. This can be seen in Figure 22.

Figure 22. CB powder is stuck in the tip of the capillary tubes in vertical dry UC cleaning.

Vertical wet cleaning in ultrasonic, carbon black powder was stuck to the bottom of the capillary tube and was resistant to come out from the tubes. To further analyze these data, the results from all the methods are summarized in Figure 23. This Multi-Vari chart displays the means for all the factors and examines the relationship between them. Individual data points are shown in grey while blue lines show the difference of %R using dry and wet capillary tubes, red lines display the differences of %R by using horizontal and vertical capillaries, and green lines illustrate the differences of %R values using cyclic VC cleaning and UC cleaning methods. According to the chart, cyclic VC gives the best results for both horizontal and vertical capillaries with DI water and 1% SLS/DI water solution. This was further analyzed with a two-sample t-test analysis and it gave a p-value < 0.0005 indicating there is a difference between these two methods.

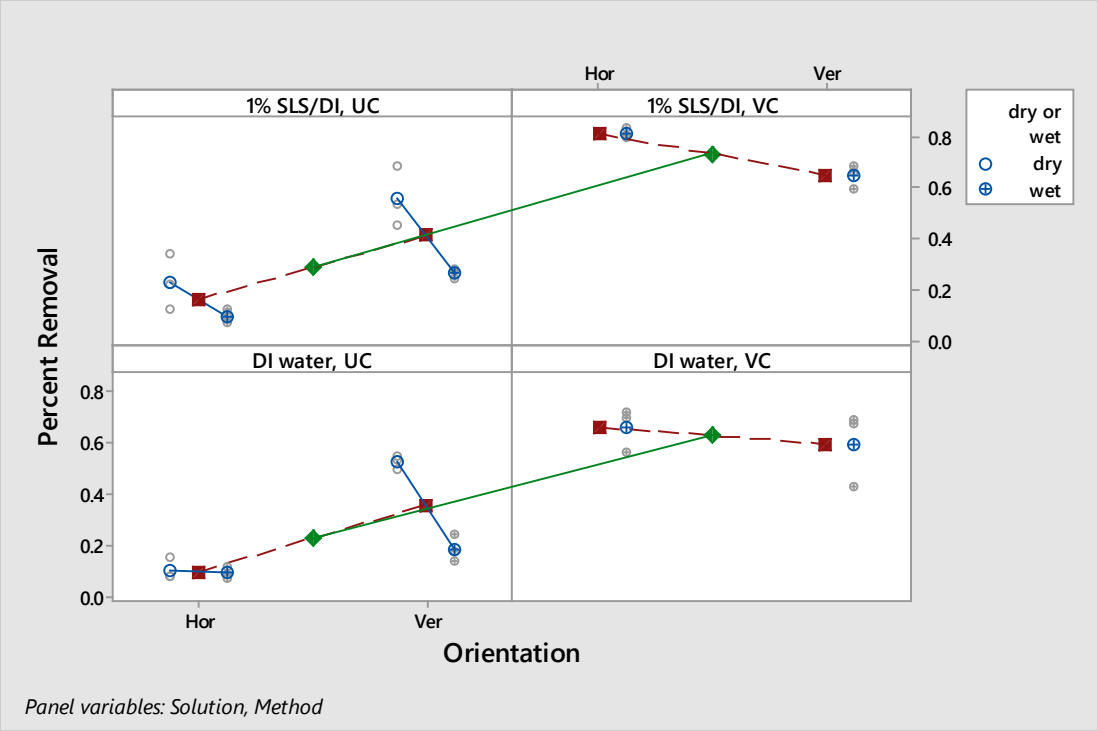

Figure 23. Multi-Vari chart for the percentage removals using cyclic VC and UC cleaning methods with dry and wet capillary tubes by changing the orientation of the tubes using different cleaning solutions.

The Interval plot for means of percentage removals with both methods is shown in Figure 24 (a). It clearly shows that means of percentage removals of both methods are statistically different. Also, the trend between the horizontal and vertical capillaries in VC cleaning and UC cleaning was studied. Two-sample t-test gave p-values of 0.078 and less than 0.0005 for VC cleaning and ultrasonic cleaning respectively (Figure 24 (b) and Figure 24 (c)). It indicates that there was less than a 7.8% chance of statistical difference in capillary tube orientation in cyclic VC cleaning. However, the orientation of capillary tubes in ultrasonic cleaning has an impact on the capillary tube cleaning efficiencies.

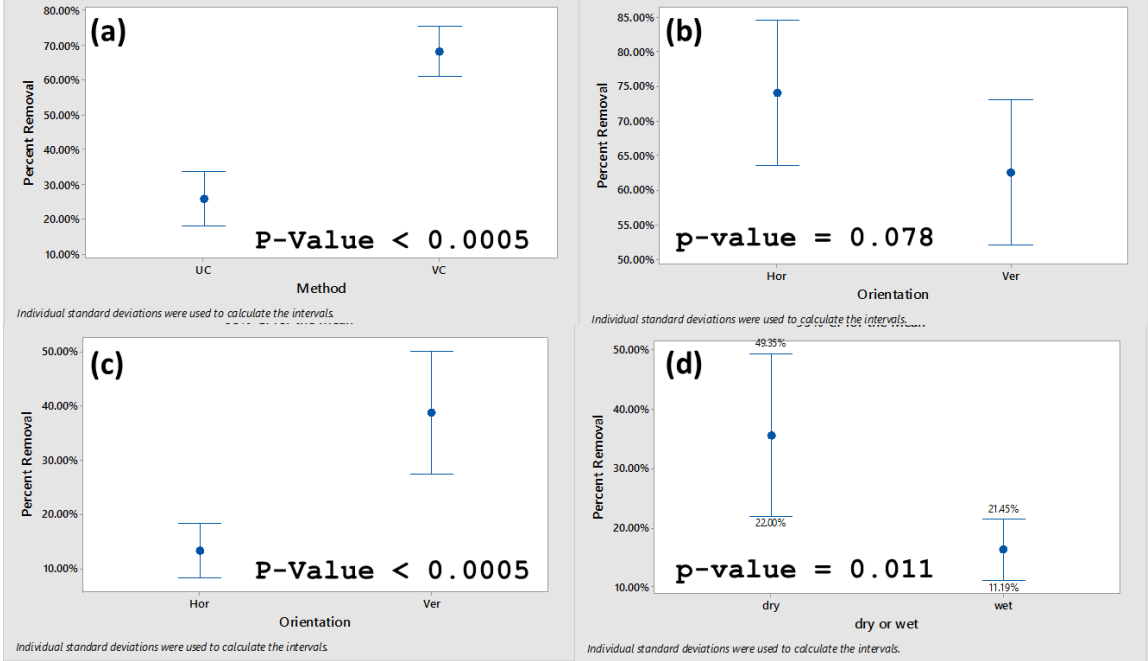

Figure 24. Mean percent removals with (a) different cleaning methods, (b) capillary tube orientation in VC cleaning, (c) capillary tube orientation in UC cleaning, and (d) dry and wet capillary tubes in UC cleaning. Note: All the experiments were done with 1% SLS/DI solution at room temperature using 15 cycles with stirring.

Since ultrasonic cleaning was studied with dry and wet capillary tubes, their cleaning efficiencies were studied. According to the interval plot in Figure 24 (d), the means of percentage removals were different for both dry and wet capillaries. Also, the two-sample t-test gave a p-value of 0.011 indicating there is a difference between these two orientations in UC cleaning.

Damage from cavitation in UC and VC cleaning methods

In UC, transient cavitation can damage the surface of the components. This effect is demonstrated using foil-covered microscope slides soiled with a sharpie. To compare cavitation damage in UC to VC at 40 °C, photographs were taken with both methods. Figure 25 shows UC and VC damage of the sharpie area on aluminum foil when cleaned

with 1% SLS/DI solution. UC experiments were conducted with different sonic power densities for 5 minutes using a 45 kHz ultrasonic bath while the VC experiment was completed with 25 cycles. In UC, there was some removal of a sharpie in sonic power density at 2.4 WL^{-1} and less damage on the foil can be seen in both trials. In UC trial 1, the less cleaning effect was seen sonic density at 7.2 WL^{-1} than 2.4 WL^{-1}. The cleaning solution which we used at 7.2 WL^{-1} was degassed not as much as the cleaning solution at 2.4 WL^{-1}. This may be the reason for the above observation since more gas in the cleaning solution causes decreased transient cavitation action.[13] Moreover, in UC trial 2, more damage and cleaning at 7.2 WL^{-1} than 2.4 WL^{-1} confirms that statement. The highest damage was observed when the samples were cleaned at 16.8 WL^{-1} in UC cleaning. As a common conclusion, if the sonic density is lowered, the transient cavitation is decreased causing low damage to the foil. On the other hand, when sonic density increases, the transient cavitation is increased causing faster cleaning action but only up to some point. Beyond that point, it may damage parts with soft metals and coatings.

There were no damage observations in the cyclic VC and some removal of sharpie was observed. There is no comparable power density rating in VC cleaning, only the number of vacuum cycles. In UC cleaning, the power level should be set to maximize the cleaning performance while minimizing the risk of damage.

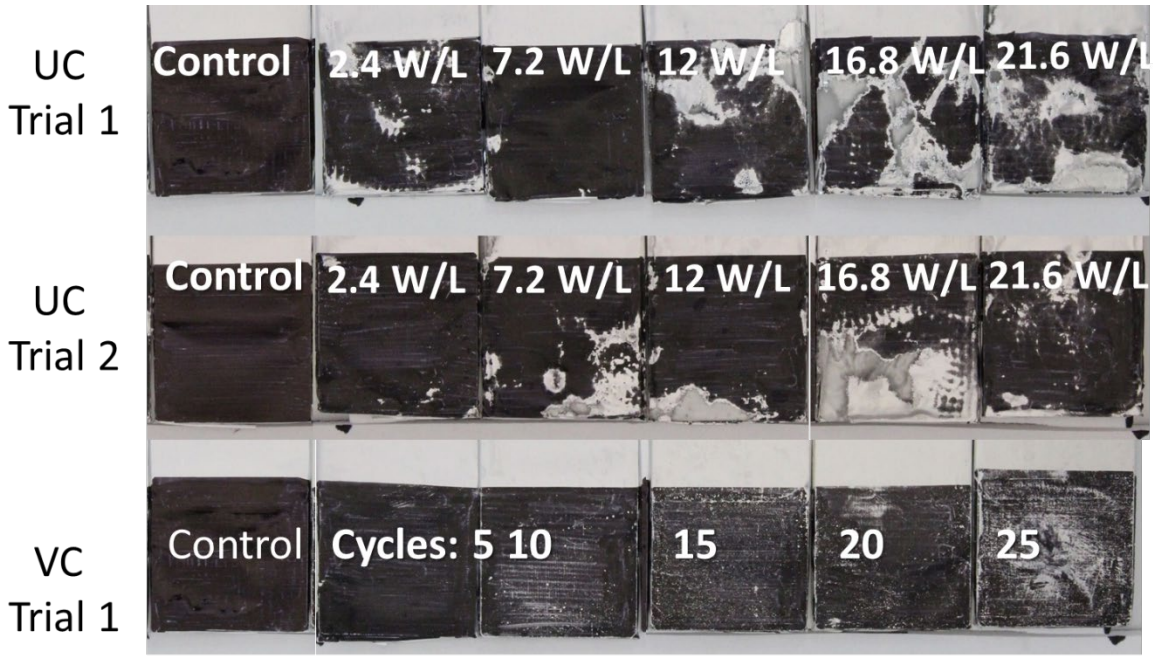

Figure 25. Damage initiated by UC on sharpie areas of foil-covered microscope slides in comparison to VC cleaning method.

Temperature effect for the cleaning efficiencies in cyclic VC

The temperature effect has been studied with the cleaning efficiencies using cyclic VC. The cleaning liquid was heated before the cleaning process. Table 5 shows that, upon the introduction of higher temperatures to the cleaning solution, higher removals of carbon black powder were observed. It can be seen that 97 ± 5% of the carbon black powder is removed from the capillaries after 15 cycles of cavity formation and dissolution in 1% SLS/DI solution at 40°C indicating that the cleaning efficiency is much higher than treatment at room temperature. By increasing the temperature, the energy of the liquid was increased and it helps to create more gas molecules with higher kinetic energies. Thus at higher temperatures, when the pressure is reduced, cavitation action is increased. Therefore, cleaning efficiencies were increased due to the increased cavity formation (nucleation) and faster cavity growth.

Table 5. Percentage removals at different temperatures.

Temperature / °C	% R
17.8	82 ± 2
25.0	84 ± 3
30.0	87 ± 5
35.0	92 ± 8
40.0	97 ± 5

Note: All the experiments were done with 1% SLS/DI water solution using 15 cycles without stirring. The ambient room temperature was 17.8°C.

Furthermore, these results were compared using multi-sample t-test analysis in Minitab 17. The multi-sample t-test is a statistical analysis designed to compare the means of multiple sets of data. All five experiments were performed using the 1% SLS/DI solution. Figure 26 shows 95% confidence intervals for the means of the data, and the overlaps between 17.8 - 25 °C and 17.8 - 30 °C indicate that there are no statistically significant differences of mean percent removals at temperatures 17.8, 25, and 30 °C. However, there is a significant difference in mean percent removals at temperatures 17.8 and 35 °C. There is an overlap between 35 and 40 °C indicating that the means of percent removals are similar at temperatures 35 and 40 °C. The p-value for 35 vs 40 °C was 0.078 showing that there is no significant difference between mean %R values at temperatures 35 and 40 °C.

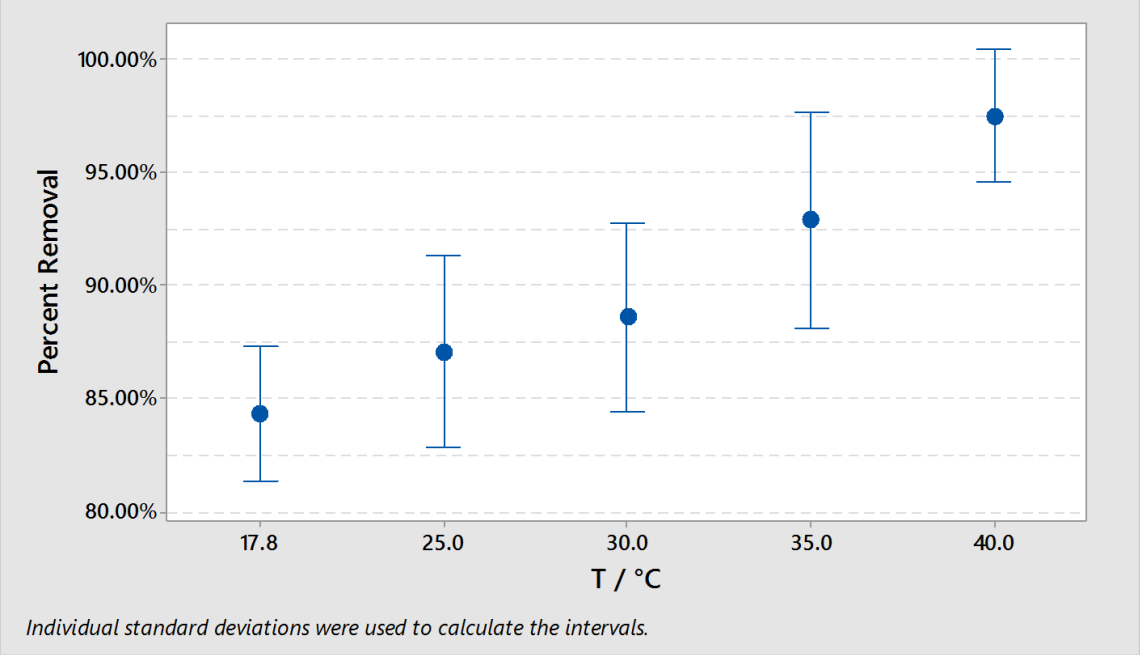

Figure 26. Multi-sample t-test analysis at different temperatures.

The stirring effect for carbon black powder percentage removals from capillaries was studied using the temperature change in the cleaning solution. In these experiments, the cleaning solution was stirred at 1200 rpm. Figure 27 shows the plot of %R data with stirring and non-stirring. The %R was determined in triplicate for all the experiments. According to this test, the carbon black powder levels in the capillaries after cleaning for both stirring and non-stirring appear to be similar. The 95% confidence intervals for the means of the data overlap significantly, indicating the means for both methods are possibly not different. The p-value of 0.266 indicates that there is no statistically significant difference in the data between these two methods. Therefore, the stirring effect did not significantly improve the cleaning of carbon black powder from capillary tubes in our studies. However, it can be seen in the videos of the experiments that stirring

the solution helps to reduce the reintroduction of the carbon black powder to the capillary tubes when the vacuum is purged.

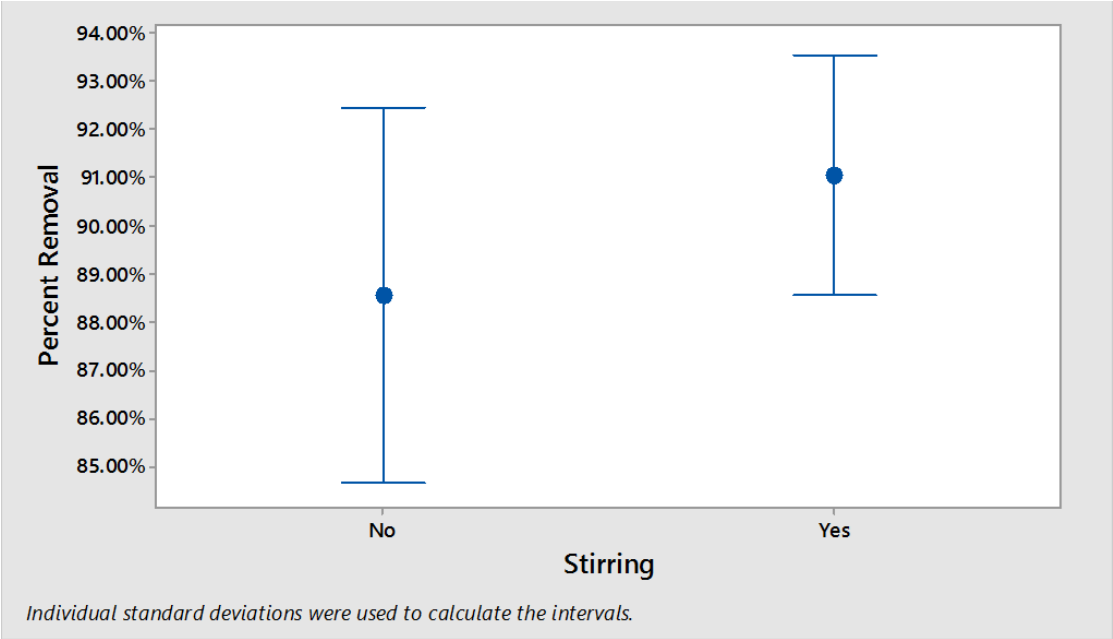

Figure 27. Two-sample t-test for stirring and non-stirring.

Surface contamination level of the capillary tubes

To find out the contamination level of capillary tubes before and after the cleaning process, the average mass of carbon black powder in capillaries was calculated. Carbon black powder contamination level before cleaning can be calculated as follows;

$$\frac{Average\ carbon\ black\ powder\ before\ cleaning}{Surface\ area\ of\ the\ inner\ cylinder} = \frac{2.053\ mg}{1.73\ cm^2} = \frac{1.187\ mg}{cm^2} = \frac{1187\ \mu g}{cm^2}$$

CB powder contamination level after cleaning can be calculated using the below equation.

$$\frac{Average\ carbon\ black\ powder\ after\ cleaning}{Surface\ area\ of\ the\ inner\ cylinder} = \frac{0.029\ mg}{1.73\ cm^2} = \frac{0.017\ mg}{cm^2} = \frac{17\ \mu g}{cm^2}$$

According to the LOD contamination level of the balance, 121 μg cm^{-2} was the smallest contamination level that can be confidently calculated using the balance. Therefore, the carbon black powder contamination level after the cleaning process is below the LOD contamination level of the five places KERN ABT120-5DM analytical balance.

Tape residue confirmation study

When capillary tubes were taped to the plexiglass windows for cleaning purposes, tape residues may influence our data. Therefore, an experiment was performed to determine if the tape residues on the cleaned capillary tubes have an impact on the percent removals of carbon black powder. The difference between means of before and after masses of capillary tubes was 0.002 mg and the standard deviation was 0.02 mg. These small values revealed that the tape residues were not interfering with the cleaned capillary tube mass in the experiments.

Optimization of cleaning in cyclic VC

From our previous studies, we figured the best temperature, number of vacuum cycles, and capillary tube orientation in the cyclic VC cleaning method. Even though stirring gave no significant difference to the results, this final study was done with stirring since it showed some improvement. When all of these successful parameters are set correctly, capillary tubes were cleaned with higher percentage removals. Table 6 shows the percentage removals of ten capillary tubes using cyclic VC cleaning at 40 °C with 1% SLS/DI solution and 15 cycles.

Table 6. Percentage removals of final confirmation study using cyclic VC cleaning.

Orientation	T / °C	Cycles	Percent Removal
Horizontal	40	15	98.1%
Horizontal	40	15	98.8%
Horizontal	40	15	98.4%
Horizontal	40	15	99.2%
Horizontal	40	15	97.3%
Vertical	40	15	99.5%
Vertical	40	15	97.8%
Vertical	40	15	99.0%
Vertical	40	15	98.4%
Vertical	40	15	99.4%

Note: 1% SLS/DI solution was used as the cleaning solution.

Average percentage removals for horizontal and vertical capillary tube orientations were 98.3 ± 0.7% and 98.8 ± 0.7% respectively. To further analyze these results, a two-sample t-test was done and the p-value of 0.326 indicates that there is no statistically significant difference in the data between these two orientations (Figure 28). This capillary tube orientation result was similar to the experiments that were done at room temperature with different orientations using the cyclic VC cleaning.

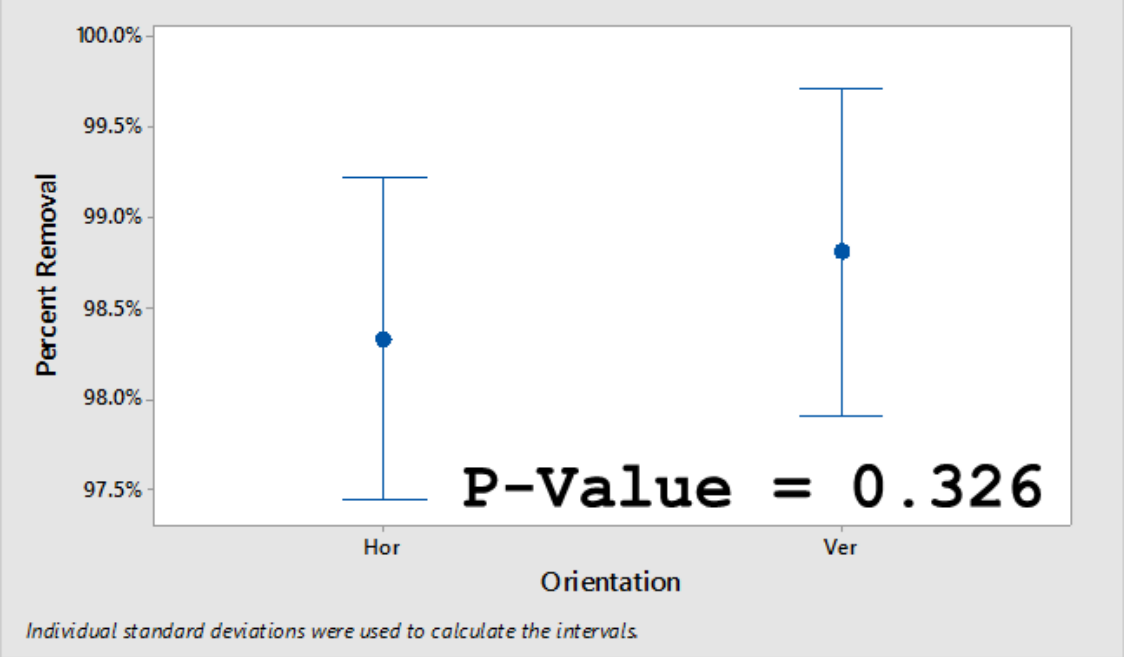

Figure 28. Interval Plot of Percent Removals using VC cleaning wth 1% SLS/DI solution using 15 cycles at 40 °C.

Therefore, considering the uncertainty of the analytical balance and the cleaned capillary tube contamination levels, we can conclude that these capillary tubes are cleaned well.

CHAPTER IV

Conclusions

The removal of micron-sized carbon black particles from capillary tubes by reducing pressure in a sealed chamber has been investigated. Vacuum cavities are generated in the laminar flow region close to the component surface which push carbon black powder soil away from the internal surface and out of the captured volume of the capillary tubes. The preliminary cleaning performance using vacuum-generated cavities coupled with deionized water removed 67 ± 7% of carbon black powder from contaminated capillary tubes. Then, minor modifications on the cleaning solution with 1% mass concentration sodium lauryl sulfate solution successfully remove 82 ± 7% of carbon black powder soil from capillary tubes with fifteen cleaning cycles at room temperature.

The carbon black powder removal performance of the newly set up cyclic vacuum cavitation cleaning is compared with ultrasonic cavitation cleaning to evaluate its cleaning efficiency. The presence of a small amount of sodium lauryl sulfate surfactant in deionized water was able to increase the cleaning efficiencies. The cleaning efficiency of 1% SLS in deionized water was able to remove approximately 99 ± 1% of the carbon black powder from capillary tubes using 25 vacuum cycles at 40 °C. When the temperature is increased in the cleaning solution, it is clear that the increased cavitation action significantly improves the cleaning efficiency. Pressure and temperature ranges in this study varied from ambient (1 bar) to 0.01 bar and 17.8 to 40 °C respectively.

The goal of testing the construction of a benchtop vacuum cavitation apparatus was achieved. This vacuum cavitation setup allowed the visualization of the cavities and

the cleaning process. The aspirator vacuum pump was an inexpensive way to generate a vacuum and it was useful since no precautions needed to be taken against the entrance of vapors into the aspirator tank. The plexiglass lid and thick windows were helpful with the visualization of the cavities inside the vacuum chamber. The combined analysis of imaging, video recordings, and gravimetric analysis tests helped to identify cleaning efficiencies in both vacuum cavitation and ultrasonic cavitation cleaning methods.

Secondly, it was found that cleaning of blind hole capillary samples was more effective with cyclic vacuum cavitation than ultrasonic cavitation cleaning at given conditions. In ultrasonic cleaning, sample orientation was important (vertical > horizontal) and dry soil seemed to be removed more effectively. The best result was 56 ± 12% for dry vertical ultrasonic cleaning with 1% SLS in DI water. Optimal operating conditions in vacuum cavitation such as 40 °C and 25 cycles can be used to accelerate the cleaning process for carbon black powder from capillary tubes. Even though stirring had no significant difference to the cleaning efficiencies in cyclic vacuum cavitation cleaning, it can be seen in the videos of the experiments that stirring the solution helps to reduce the reintroduction of the Carbon black powder to the capillary tubes when the vacuum is purged. This study has shown experimental evidence for cleaning capillary tubes using cyclic vacuum cavitation coupled with a dilute surfactant solution as a good cleaning approach.

CHAPTER V

Future Studies

As cleaning with vacuum-generated cavities has a large potential to improve, continuous investigations can be done to improve the cleaning efficiency. Future studies should be done in the characterization of component surfaces to determine the effect of cleaning action on its structure such as damage to the surface of the components.

Weighting multiple soiled capillary tube samples in a batch can be done to get a lower LOD from the 5-place balance. To achieve nanoscale contamination levels, more sensitive analytical techniques on soil extracts from the capillaries can be used for cleanliness monitoring. However, these techniques would have difficulty with insoluble carbon black powder.

Since ethanol and acetone have higher vapor pressures than water at room temperature, cavitation should be much more rapid with these solvents. Also, the ability to interact with oily soils may be much more favorable with these volatile solvents. Therefore, in the future, it is worth studying carbon black powder removal using ethanol and acetone since they may be able to clean the carbon black powder from the capillary tubes much faster and would be much faster drying.

It may be beneficial to study ultrasonic cavitation followed by vacuum cavitation to see if effective cleaning can be achieved at lower temperatures. Transient ultrasonic cavitation can loosen the soil from the surface but has difficulty flushing the soil from deep blind holes. Vacuum cavitation is effective at flushing these structures.

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EDUCATION

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PRESENTATIONS

1. Ariyarathna. N.; Volek. T.; Williams. D.L. Comparison of vacuum cycle cavitation to ultrasonic cavitation cleaning applications, American Chemical Society (ACS) (2021)

2. Ariyarathna. N.; Jayawardana. Y.; Pathmanathan. R.; Weerasooriya. R. Geochemical classification of groundwater by piper diagrams and principle component analysis, Water and Development Congress & Exhibition, Colombo, Sri Lanka (2019)

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4. Ariyarathna. N.; Weerasooriya. R. Applications of Integrated Water Quality Index (IWQI) for rapid demarcation of palatability problems in groundwater, Geological Society of Sri Lanka (2019)

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- Membership - American Chemical Society (ACS) (2019 – present)
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- Membership - Rotaract Club, University of Peradeniya (2017 - 2019)
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